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DESULPHURISATION OF SHALE OILS,
OIL-SHALE RESOURCES,
and
PRODUCTION OF OIL FROM CANNEL COAL
AND ALLIED MINERALS.

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THE INSTITUTION OF PETROLEUM TECHNOLOGISTS.

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Issue of Journal. The Journal appears in four parts per sessional volume, viz. in December, February, April and June. A brochure describing the origin, progress and purposes of the Institution, and comprising also the Memorandum and Articles of Association, the By-Laws and Regulations of the Institution, the Library Catalogue to date (with subject index), and the List of Members, was published in September, 1915, to be followed annually by revisions of the Library Catalogue and List of Members.

Members of all classes are entitled to receive these publications free: for additional copies of the Journal they will be charged at the price of five shillings per part, and of the brochures at the prices stated on the wrappers, varying in proportion to bulk.

Changes of Address. It is particularly requested that members notify the Secretary immediately of any change of address; and members are also requested to advise the Parcel-Post Department as well as the Letter Office, of any temporary change of address, as, unless this is done, parcel-post packets will not be re-addressed; but will be returned to the offices of the Institution in London, thus incurring further expense for postage.

To Authors of Papers. Papers should be written in the third person, and the copy should be carefully corrected by the author before it is presented.

All drawings, diagrams or other illustrations should be sent in a fit state for direct photographic reproduction.

All quotations, technical terms, and localisms should be indicated by means of inverted commas.

Foreign weights, measures, and costs should be given whenever possible; and also their English equivalents.

It is suggested that authors append as complete a bibliography of the subject treated as may be possible.

All papers and notes submitted to the Institution are supposed to be original communications unless distinctly stated to be otherwise, in which case the exact reference to the previous publication should be given.

Communications upon papers read at meetings, notices of personal movements, or other matter for which publication is desired in the next issue of the Journal, should be in the hands of the Editor on or before the last day of January, March, May or November as the case may be. Subsequent delivery may be too late for insertion.

Advertisements in the Journal. A limited number of Advertisements of firms interested in the Petroleum Industry may be inserted in the Journal. Application for terms, etc., should be made to the Secretary.

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PERSONAL NOTES OF MEMBERS AND SPECIAL NOTICES.

It is suggested that Members of the Institution send information regarding their movements to the Secretary for insertion under this heading.

We regret to record the death of another of the founders of our Institution in the person of Mr. Alexander Milne, who in January last closed a career of more than 75 years of important activity.

Born at Forfar in 1843, and selecting flour-milling as his life work, he left home in 1863 to take charge of some New Zealand mills. Unfortunately these mills had been destroyed by floods before his arrival, and in his subsequent travels he landed in Valparaiso, from which centre he first organised Chilean milling business, and afterwards developed a similar very successful Peruvian enterprise.

Later, he became the pioneer of the Peruvian petroleum industry on modern lines, and was also engaged in extensive sugar production. Mr. Milne was practically the discoverer of the valuable Lobitos Oilfield, and it is chiefly due to his energetic and far-seeing management of that field that its present prosperous condition has been brought about.

Mr. Milne was a man of singularly attractive personality and his loss will be keenly felt by his many friends in this country and in South America.

Major W. H. Fordham, R.F.A., is proceeding to Galicia with the Central European Military Mission.

Mr. W. A. Guthrie has received the honour of the O.B.E., for the part he took in the destruction of the Rumanian oil wells and refineries in November and December, 1916, to avoid their falling into the hands of the enemy. He is now with the Egyptian Government as Petroleum Research Chemist.

Mr. C. B. Rosenplaenter returned from Algeria in February.

EDITORIAL.

Indexing. The impossibility to the most highly-trained mind of retaining in memory all the minutiæ relative even to a special subject of study, not to speak of allied or more remote topics, necessitates not merely accessibility of trustworthy sources of information, but that such access shall be available promptly, if not on the instant of requirement. Every student knows how liable are questions suspended for future reference to remain in that state, unless the point is one of too pressing importance to be thus left open.

In many cases the reference would involve no more time than opening the requisite volume, turning up an indicated page, and noting the desired particulars. The strenuous conditions now obtaining render exactitude the more essential, and by precluding in most cases the formation (and care) of an adequate private library, leave only the alternative courses of conveying the question by letter (or messenger) or of personal visit to the source of information. For the former procedure one must remember the adage: "If you want a thing done, go: if not, send," and reverse the legal maxim into "who acts through another, acts not."

In any case an essential point is to know where to get the desired detail. The Society of Chemical Industry published in 1918 four several articles on filing and indexing (vol. xxxviii, pp. 67, 317, 360, 377). Each writer assumed the possession of the requisite knowledge in print or manuscript, some propounding schemes for extensive libraries, others for small private collections of text or abstract. It is evident that any system must be modified to meet the special conditions, and that the method of filing or shelving is a secondary consideration to that of indexing in such a way as to involve the least addition to the student's work. Attention to scientific detail should be diverted as little as may be to mechanical search in index or shelf.

It may also be laid down as undeniable that complete reliance cannot be placed upon abstracts, in which the precise point now raised has generally been ignored by the abstractor. Reference to the original text must therefore be furnished by the index consulted. If this is of private ownership, it can bear indication by chosen conventions, as to which works are on the proprietor's own shelves,

instantly accessible, and which are to be sought in other libraries. Shelf-numbers or other classificatory data can be added, but these are liable to changes in all "live" libraries.

A rather crucial question in indexing is that of alternative synonyms, which may occur indifferently to the user, or even to the compiler at different times, the equivalent being momentarily forgotten. There are but few subjects which have no such synonym, and cross-references are essential. If some entries are placed under each, the user may be misled into taking the part for the whole, before the mistake is detected and all placed under the preferred synonym. Choice should be made early, and the requisite cross-references inserted at once.

Judgment must also be exercised as to intermediate subdivisions, not expressed in titles. Thus a reference to Maikop should be indexed under Kuban and Russia, whether those names occur in the title or not; but an article on Sterlitamak does not call for entry under Ufa, as that province has no hydrocarbons except in the Sterlitamak district, in contrast with the continuous oil belts of the Kuban Territory.

Skill in indexing is not a difficult art, but requires practice, and we would recommend all whose surroundings admit, to take up the compilation of the bibliography of some topic of interest, and of a subject-index thereto, advancing simultaneously. Slips of paper about $2\frac{1}{2} \times 8\frac{1}{4}$ inches can be obtained by the ream for a few pence more than the folio sheets from which they are cut (this size is 10 slips from a sheet of "cap."). There is an advantage in procuring a quantity at once, that they range neatly in drawers. "Guides" at each initial or other subdivision may be of strawboard, either of the same size, merely making a perceptible break, or a quarter of an inch higher, to bear letters or words, visible above the mass of the slips.

Besides its supreme utility, bibliography is, literally as well as metaphorically, a pursuit that becomes the more entrancing the longer it is followed.

Thirty-Fourth General Meeting.

This Meeting was held at the House of the Royal Society of Arts, on Tuesday, 17th December, 1918, Mr. Charles Greenway (President) occupying the Chair.

The Members of Council present were: Captain R. W. Barnett, Mr. H. Barringer, Prof. J. S. S. Brame, Dr. A. E. Dunstan, Mr. Arthur W. Eastlake, Dr. W. R. Ormandy, and Mr. T. C. Palmer. A letter of regret for unavoidable absence was received from Prof. Sir John Cadman.

The Production of Oil from Mineral Sources.

By Dr. F. Mollwo Perkin, Ph.D., F.I.C., F.C.S., *Member*.

Many and very various products can be obtained by the carbonisation of bituminous minerals, the character and quality of the materials produced depending mainly upon the temperature at which the process is conducted. It does not, however, follow that all identical products will be obtained from different bituminous materials when they are subjected to the same temperature conditions, because the composition varies, and consequently when subjected to heat the method of decomposition varies. The organic body or bodies in shale are called kerogen and this, on being subjected to moderate heat, yields oil of the olefine and paraffin series, ammonia also being produced. The organic matter in coals and cannels is generally described as volatile matter, and probably differs considerably in chemical character from the kerogen; consequently on being subjected to moderate heat different products are obtained, although they also are mainly of the olefine and paraffin series. When coals are subjected to high temperatures, a different class of hydrocarbon is produced, mainly of the benzene series. It is probable that this would also be the case to a greater or less extent if shale were also subjected to high temperature in retorts similar to those employed for heating coal.

It is extremely difficult to obtain an even distribution of heat in any carbonisation process; consequently it is by no means easy to make certain of always obtaining the same products from a given material in the same proportions, and great care has to be exercised to ensure that the conditions are as nearly constant as possible. The form of the retort has much to do with the quantity and quality of the products obtained. In all cases the first effect of the heat is low-temperature distillation, because the material is introduced cold into the retort, and however high the temperature

of the retort, the heat must first get through the badly-conducting mass before the temperatures can approximate to that of the retort, and by that time a considerable part of the volatile matter will have been driven off. Then another question arises, i.e. whether the form of the retort is such that the volatile products, as they are formed, come in contact with the surface of the retort before being drawn off, or whether they are removed without being further heated after they have been expelled from the material. The quality and character of the final products depends almost entirely upon this. Thus in horizontal gas retorts the volatile matter as it leaves the coal comes in contact with the highly-heated arch and sides of the retort, before it enters the ascension pipes and is carried to the hydraulic main. This causes radical changes in the volatile products, and hydrocarbons of the benzenoid or aromatic series are largely produced. On the other hand, in a vertical retort, the volatile products, as they are released from the coal, ascend upwards through the cold incoming coal, only a portion coming in contact with the hot walls of the retort, and as a consequence the resulting products contain a considerable proportion of hydrocarbons of the aliphatic series.

As already mentioned it is probable that in all methods of carbonisation the actual volatilisation takes place at a comparatively low temperature, the final product being obtained by a further chemical or physical action, depending upon the design of the retort and the temperature to which it is heated.

The first action takes place at a very low temperature, and is the removal of occluded gases, the second is the volatilisation of the hydrocarbons of the coal itself, but to say when the one ceases and the other commences is a very difficult matter. There is also the question of speed of reaction; if a bituminous material is kept at a moderate temperature for a long time, practically the whole of the volatile matter can be removed, but the products produced vary with the time. Thomas (*Journ. Soc. Chem. Ind.* 144, 1876) found that when coal is submitted to prolonged action of heat in a vacuum, the quantity of gas produced increases with the time, and the quality also varies as the reaction progresses. It must be borne in mind that the products produced by the destructive distillation of bituminous material were not there in the conditions in which they are finally obtained. By extraction with solvents we probably obtain them as they exist in the mineral, but the moment we try to distil them and to remove the solvent decomposition and polymerism take place. These changes are very much more marked in certain carbonising retorts than in others,

and the higher temperature the greater the decomposition. For example, in horizontal gas-retorts, where the volatile materials as they are driven off come into contact with the highly-heated surface of the retort, they are further decomposed, and as a result a smaller yield of tar is obtained than with a vertical retort where, as already mentioned, the volatile products pass up through the ascending cold material, and only a portion strikes against the highly heated retort walls. Furthermore, so thorough is the decomposition in horizontal gas-retorts that complete splitting up of the hydrocarbon results, and carbon is deposited on the roof and sides of the retort, whilst the tar itself contains more free carbon than that obtained with the vertical retorts, and in smaller yield. Complete decomposition also takes place to some extent in vertical retorts, and some carbon is formed on the heated walls, but less than in the case of the horizontal retorts so that the tar-yield is higher. On the other hand with retorts heated to moderate temperature, say 450° to 500° C., against the 1,100° C. of the horizontal and the 1,300° to 1,400° C. of the vertical, no carbon is deposited on the sides of the retort, and the amount of free amorphous carbon in the tar is very small and a maximum yield of tar (crude oil) is secured.

Whether high or low temperature should be employed for carbonising bituminous material entirely depends upon what products are required. For gas works, where a large volume yield of gas is required, high temperature is essential, but where motor-spirit, fuel oil, lubricating oil and paraffin wax are required, low-temperature carbonisation must be adopted. In low-temperature carbonisation the gas produced is less than half that obtained by high-temperature carbonisation, and contains less hydrogen and more hydrocarbons than the latter; low-temperatures carbonisation could therefore not be employed for the manufacture of gas for lighting purposes. The main distinctions between high and low-temperature are as follows :—

	High Temperature.	*Low Temperature.*
A.	Large volume of gas, say 12,000 c.f. on the average.	Low volume of gas, say 5,000 c.f. on the average.
B.	Yield of sulphate of ammonia, on average, say 20 lbs.	Yield of sulphate of ammonia on average say 10 lbs.
C.	Yield of tar on average, say 11 gals. per ton of coal carbonised.	Yield of tar (crude oil) on average, say 20 gals. per ton of coal carbonised.

	High Temperature.	*Low Temperature.*
D.	Tar is largely of aromatic series and yields benzol, toluol, naphthalene, anthracene, carbolic acid, cresols. These products are the raw products for the manufacture of dyes, explosives, photographic chemicals, drugs and many other synthetic products.	Tar crude oil consists of hydrocarbons of the aliphatic series (paraffins, olefines and naphthenes). From the tar can be obtained motor spirit, fuel oil, lubricating oil and paraffin wax. The tar acids are useful for disinfectants, but are of no use as raw products for other industrial purposes.
E.	Residue a hard coke with little volatile matter, useful for industrial purposes.	Residue a soft coke with 8–10 % of volatile matter, a good smokeless household fuel.

It should be mentioned that when coals high in volatile elements and rich cannels are subjected to low-temperature distillation, much larger yields of crude oil are obtained, as much as 40 and 60 gallons. (It is usual to use the term crude oil and not tar when speaking of the first product obtained by carbonising at low temperature.)

Oils obtained by the carbonisation of bituminous material come under the same category as natural oils; they may therefore be classed as mineral oils, even although their origin was probably organic as was that of natural oils, but many organic substances, such for example as peat or wood, will give oils of a similar character when carbonised under suitable conditions. Later on I shall refer to the carbonisation of peat.

At the outbreak of the war, the world's production of natural oil was in the neighbourhood of 50,000,000 tons, and last year over 60,000,000 tons.

Before the war Great Britain was, with the exception of the oil obtained from the Scottish Shale Oil Industry (275,000 tons crude oil), entirely dependent upon imported oil for all the various purposes for which oil is required. Our aeroplanes, warships, motor cars, etc., were dependent upon sea-transport for petrol and fuel oil, and our machinery for lubricants. Unfortunately we are still in the same position. Great Britain, with her vast Navy and her great fleet of aeroplanes, is dependent for fuel upon sea-borne transport, and this should not be necessary, or at any rate only in a partial degree. In the future our merchant ships will probably burn oil fuel. We have at hand mineral resources from which we can produce mineral oils—petrol, fuel oil, lubricating oils and paraffin wax. Why do we not employ these sources? There are of course many difficulties in founding a new industry, and

one of the greatest difficulties has been Government action or inaction. There is now, however, a stirring among the dead bones, a great deal of experimental work has been carried out, much of it on semi-commercial plant, and there are now several large schemes under consideration, which would involve the putting up of an extensive plant both for retorting and refining the oil, and to obtain power from the residuals, or domestic fuel.

Historically the first low-temperature distillation process was devised by James Young in 1847, who had his attention drawn to oil which flowed from some coal workings in Derbyshire.

From this oil Young succeeded in abstracting, on a commercial scale, wax and burning oil and a heavy lubricating oil. As, however, the supply of raw oil soon became exhausted, Dr. Young, believing the oil to have been produced by *low temperature distillation of coal by terrestrial heat*, endeavoured to obtain oil by low temperature distillation of coal in retorts. He experimented with a large number of English and Scottish coals, and finally found that the Torbanehill or Boghead coal in West Lothian yielded from 120 to 130 gallons of oil per ton of coal distilled. In 1850, Young took out a patent for "obtaining paraffin oil, or an oil containing paraffin, from bituminous coal." By the end of the same year the Bathgate Oil Works was started under the name of Young, Meldrum and Binny. The Torbanehill deposits were worked for about twelve years, when they became exhausted, and since that time the Scottish shales of Linlithgow and Midlothian have been the principal source of supply.

It is interesting to note that Young's process was largely worked in America until the advent of natural oil in 1859, and the accompanying oil fever, when the distillation of shale was discontinued.

The quantity of oil and of sulphate of ammonia yielded by shales varies considerably. It may be as high as 30 to 40 gallons per ton of shale, but is sometimes as low as 17 to 18 gallons. The original shales worked by Young yielded from 40 to 45 gallons per ton, and with present-day methods of retorting would undoubtedly have yielded more. The average yield in Scotland to-day is, however, only 23 gallons per ton. The amount of sulphate of ammonia obtained depends largely upon the percentage of nitrogen in the original shale.

Young's patent expired in 1864, and this led to a great expansion in the Scottish oil industry; but shortly afterwards, owing to the output of natural oil and the American competition, it experienced a severe check. The discovery in 1859 by E. L. Drake of oil wells in Pennsylvania was followed by the importation into

England of lamp oils. In the early days of the shale-oil industry lamp oil was the principal product, and during the run of Young's patent fetched about 2s. 6d. per gallon. After the expiry of the patent it fell, owing to competition, to 1s. 6d., and when the American oil came on to the market, the price dropped much lower, until before the war illuminating oil could be obtained for about 5d. per gallon, and the price has even been lower.

The amount of shale treated in the Scottish retorts is over 3,000,000 tons per annum, which before the war produced about 275,000 tons of crude oil, or over 70,000,000 gallons. I do not know how much was produced during the war. The marketable products produced by the distillation and refining of the crude oil and their approximate yields are on a pre-war basis:—

Motor spirit	600,000 gallons.
Naphtha	4,400,000 „
Burning oil	20,000,000 „
Gas or fuel oils	12,000,000 „
Lubricating oils	10,000,000 „
Paraffin wax	25,000 tons.
Sulphate of ammonia	55,000 „

The total value of the products exceeded £2,000,000 per annum.

The first process after mining is to pass the shale through a breaker, where it is converted into pieces about 6 in. square. It is then discharged into iron tubs, which are run along rails, and discharged into the retorts.

The Bryson retort generally used is long and narrow. There is an iron table below for supporting the shale, and a revolving arm to cause the shale to move downwards.

Through the centre of the table a steel spindle or shaft projects, on the upper end of which is a curved arm, and this, when rotated, pushes some of the shale off, causing it to fall over the edge of the table into the hopper below. The shaft carrying the curved arm passes through a stuffing-box on the hopper, and has a ratchet and lever fitted to the lower end actuated by a bar or rod of T-iron, which is made to travel horizontally, and is driven by gearing from a small electric motor. The motion is comparatively slow, the arm making only one revolution in twenty minutes, and the action is quite simple, the throughput of shale being regulated at will. The upper portion of the retort is of cast-iron, 11 ft. long by 2 ft. in diameter at the top, enlarged to 2 ft. 4 in. at the bottom. The lower part is built of firebrick, and is about 20 ft. long, enlarged to 3 ft. at the bottom. Heat is applied externally from uncondensable gases obtained from the distillation of the shale (assisted,

in the case of the poorer qualities of shale, by producer gas), and is made to circulate round the retort by chequers. The heating gas enters near the bottom of the brick portion of the retort, along with a certain quantity of air, and a high temperature (650° C. to 900° C.) is maintained in this portion, where the nitrogen of the shale is converted into ammonia, which is aided by a continuous supply of steam delivered at a slight pressure into the bottom of the hopper. The oil vapours and gases are distilled from the shale in the cast-iron portion at a temperature of about 500° C., and, along with the ammonia gas, are drawn off by the exhausters, at a branch pipe at the top of the cast-iron retort, through the atmospheric condensers. The condensed liquid oil and water containing ammonia flow into a small separating tank. The gases then pass through ammonia scrubbers in which they are " washed " for ammonia, then through the naphtha scrubbers where the lighter gases, which could not be caught in the atmospheric condensers, are scrubbed with oil, and a good quality of light oil or naphtha is recovered. The uncondensable portion passing from these scrubbers is burned under the retorts. Shales of average quality yield sufficient gas, not only to heat the retort, but also to burn under the steam boilers.

The ammonia water got from the atmospheric condensers is pumped through a heater, in which it is raised in temperature by the spent or waste water flowing from the still, and passes into the top of the still, which is circular in shape, about 80 ft. high and has a series of cast-iron shelves fixed horizontally every 2 ft. or thereby from the top to near the bottom. Steam is injected into the bottom of the still, and passes to the top carrying with it the volatile ammonia, while the water, after traversing the whole area of each tray, passes down by a seal pipe from one to the other, and flows out at the bottom as spent water into a concrete tank containing a cast-iron worm, which acts as a preheater for the ammonia water on its way to the still. During its progress from the top to the bottom of the still, the water is diverted into a chamber containing milk-of-lime, which sets free the fixed ammonia.

The ammonia as it is liberated from the still is passed into a lead-lined saturator containing sulphuric acid, where it is converted into sulphate of ammonia. As showing the care which is exercised to prevent waste, the exhaust steam and waste gases from the saturator are passed into the retorts and utilised to aid in the formation of ammonia from the shale.

Refining the Oils.

The oil as condensed in the coolers is a black, semi-solid liquid. It is pumped into overhead tanks, which are so arranged that the stills can be fed by gravitation. Before being run into the still, the oil is allowed to settle from twelve to eighteen hours, being kept sufficiently warm for it to remain quite fluid, so that the water may the more readily settle. The oil is then fed into the stills and fractionated.

In the retorting of shale, ammonia is one of the most important products produced, and the retort is specially designed to obtain the maximum yield both of oil and ammonia; the oil being produced in the upper cooler portion of the retort and the ammonia in the lower hotter portion, where the shale which has been freed from the main portion of the volatile matter is acted upon with super-heated steam.

As far as the production of oil is concerned the retorting of shale is a low-temperature process but mediumly high for the production of ammonia.

The residue from shale which remains after the shale has been freed of its volatile matter and acted upon by a superheated steam contains no free carbon and consequently is valueless as a fuel in that it contains no combustible matter, and unfortunately the mineral residue is also valueless as is witnessed by the enormous dump heaps to be seen wherever shale is retorted.

The ordinary shale retort is not adapted for dealing with caking bituminous material nor indeed for treating cannel and non-caking coals, hence the larger amount of research work which has been carried out in the endeavour to devise a suitable retort for dealing with bituminous material which contains a large amount of fixed carbon, and will yield after extraction of the volatile matter a good fuel for domestic purposes. It might be replied, "but this is already obtained in the gas works where, when coal is carbonised to produce gas, a residue of from 68 to 70 per cent. remains in the form of coke, and beside which tar and ammonia are produced." True, coke is produced and this coke contains a very low percentage of volatile matter and for this reason is not adapted for burning in the ordinary grate. Coke produced by low-temperature carbonisation (350°–550° C.) is softer than that produced at high temperatures (above 900° C.) and usually contains from 7 to 11 per cent. of volatile matter. The presence of this volatile matter causes the coke to burn readily, practically without flame or smoke and to give out a great heat. It is consequently very clean for

household purposes, and if it were used instead of coal the cost for painting and decoration of the house would be considerably reduced. Furthermore, the atmospheres of our large towns and cities would be very much less contaminated by smoke and the living conditions would be healthier.

In producing this smokeless fuel by low-temperature carbonisation there are produced at the same time oils of the aliphatic series, which on refining yield motor-spirit, fuel oil for internal-combustion engines or for direct boiler-firing, lubricating oils and paraffin wax, beside which there is a small quantity of ammonia, and sufficient gas to fire the retorts, and leave a small surplus.

Now, in low-temperature practice there are two possibilities, either of which might be a financial success, or they might be combined.

1. The production of oil and smokeless fuel.
2. The production of oil and the conversion of the fuel residue into power-gas by gassifying it in a producer.
3. The production of oil, using a portion of the fuel for domestic purposes and gassifying the remainder.

In districts where power is not required for manufacturing purposes but where coal or cannel could readily be obtained without having to transport it for long distances then the 1st proposition would be the one to embark on.

On the other hand where cheap power in large bulk is required then No. 2 would be the process to take up. Probably in all cases a certain proportion of the residue would be sold as smokeless fuel.

I think it again necessary to meet an objection which is sometimes raised. "If you want power why not gassify the coke produced in the gas works in a producer?" Well, there are uses for coke as produced by the gas works and there is this difficulty in gassifying it in producers, that coke produced at high temperatures and containing very little volatile matter only yields up a small proportion of its nitrogen in the form of ammonia. A good yield of gas, however, is obtained.

I will give examples taken from actual practice. In each case about 50 tons of coke was gassified.

1. Coke produced on low-temperature plant—the plant of the Tarless Fuel Syndicate—the analysis being:—

Volatile Matter	9·87	Nitrogen.. ..	2·05
Fixed Carbon	74·54	Ash	12·59
Yield of gas per ton gassified			128,000 c.f.
B.Th.U. of gas			127
Sulphate of Ammonia			101 lbs.

2. A coke produced on the Glover-West retorts, where the temperature of the flues was about 1,400°C, consisted of

Volatile Matter	1·31	Nitrogen.. ..	1·52
Fixed Carbon	86·90	Ash	9·27

Yield of gas per ton gassified .. .. 175,000 c.f.
B.Th.U. of gas 132
Sulphate of Ammonia .. .. 23 lbs.

These two results show that the amount of sulphate obtained was much larger with the low-temperature coke than with the high-temperature coke.

On the other hand it must be borne in mind that in gas practice more than double the quantity of gas of high calorific value is produced than by low-temperature carbonisation and the initial yield of sulphate is more than double that obtained in low-temperature practice.

I wish again to insist on the absolute difference in the products obtained by carbonising at high and at low temperature. This insistence is necessary because I have frequently heard it remarked that the tar or crude oil obtained by low-temperature carbonising can be used for making dyes, etc. I have even heard those engaged in producing this crude oil make this remark, followed by saying: "If we go in for this process we shall be able to win the dye industry back from Germany." The Coalite Co. claim their process to be a low-temperature process, and then state that they produce more benzol and toluol than is produced in high-temperature gas practice. All I can say is that if they produce benzol and toluol, either their process is a high-temperature process, or else they treat the low-temperature oil by some cracking process. If, however, they require benzol and toluol, why produce it in two stages?

I remarked above that those who do not know the quality of the low-temperature products have said: "We shall then be able to win back the dye industry from Germany." We nearly lost the war to Germany from lack of oil. Lord Curzon recently told us that at one time there was only a stock of 900,000 tons in the country against a minimum of 1,500,000 which the Admiralty considered necessary. The Fleet, in fact, had to restrict its exercises in order that if a battle took place there should be sufficient oil to go round. I venture to say that when we know more of the internal management of Germany during the war it will be found that they produced large quantities of oil by low-temperature carbonisation of bituminous material. They were doing so before the war.

I believe that one of the reasons why low-temperature carbonisation has—so to say—hung fire, is due to the exaggerated claims

made by inventors backed up by Company promoters. In all distillations of bituminous material water is obtained along with the oil and is at times extremely difficult to separate as the specific gravity of the crude oil approximates to that of water. In fact it is not unusual to find 30 to 40 per cent. of water in the crude oil. This has all been lumped in as oil, hence the impossible claims for oil yields which have been made.

The days of exaggeration are, I trust, passed, and careful research has taken the place of romance. If the claims are more moderate at any rate we are working on a sure foundation, and many of us believe that a home oil industry can be founded on business lines, which, although not rendering us self-supporting in oil will, at any rate, supply a portion of our needs and tend to prevent foreign dictation of prices.

Now let us briefly examine the minerals and sources from which oils may be obtained. Excluding Scottish shale, which is at present being so successfully worked, and which I have already dealt with, we have the unworked Norfolk and Dorset Kimmeridge shales, of which there are very large beds. Unfortunately, however, oil obtained by retorting Kimmeridge shale, although the yield is good, is of little value owing to its high sulphur-content, 6–8 %. Until some satisfactory and cheap process is devised for desulphurising either the shale during retorting, or the oil on refining, there is not much likelihood of oil in quantity being obtained from this source. There are large and thick seams of lignite and brown coal in Devonshire. These lignites in the Bovey Valley occupy an area of probably from 12 to 15 sq. miles, and outcrop at Bovey Tracey. They were known as far back as 1689, and the outcrops were worked in 1760, and used locally as domestic fuel. The lignite, however, is not a satisfactory fuel, owing to its unpleasant odour when burning and its high percentage of ash ; consequently, when ordinary coal became available, the employment of lignite for domestic purposes died out.

Before the war a Company was formed to work these deposits, and to ascertain whether the material would be suitable for power purposes, and for supplying the neighbouring towns and villages with electric light.

According to information which has been given to me, an electrical power station of 100 h.p. was erected, and two vertical engines driving three-phase generators were worked from gas made from the lignite by burning it in a producer. It is also claimed that at that time—i.e. before the war—the lignite could be delivered to the producer at 2s. 6d. per ton. As the average consumption of lignite

was 3·4 lbs. per brake h.p. per hour, power could be obtained at 0·06 of a penny per B.O.T. unit. Although this has no direct bearing on oil, I mention it here because with ordinary coal the average fuel-consumption is about 2 lbs. per b.h.p.h., and before the war the average cost of coal at the pit-head might be taken at 9s. to 10s. per ton.

As to the oil obtained from this lignite, when retorted, the best results gave from 33 to 35 galls. of crude oil per ton of air-dried material retorted. The coke remaining was tested on a suction-gas plant with very satisfactory results.

There is one point to which I wish to draw attention. It has been generally supposed that the Company which carried out the tests was a German Company—in fact, I was so informed, but incorrectly. The Company was English, but they employed German chemists and drillers because it was thought they would be better and cheaper than the native material. As to cost we do not wish to sweat our workmen. The war has shown that British chemists are as good and resourceful as the Germans, and the engineers better.

There is one other point which should be mentioned. The lignites and brown coals contain certain proportions of a hard wax called montan wax, and before the war this was obtained exclusively from Germany. This wax is valuable on account of its high melting-point, and is used in the manufacture of boot and other polishes. On behalf of the Chiswick Polish Co. I examined a number of samples for this product; the amount obtained, however, did not exceed between 7–8 %. It would not therefore pay to work the deposits for montan wax alone, but if the wax was first extracted, the material then retorted for oil, and the residue finally passed through a producer for power purposes, the proposition takes on a different complexion.

Before dealing with the largest source of mineral from which oil can be obtained—viz. coal—I will briefly mention the possibilities of utilising peat. We have enormous deposits in the United Kingdom and Ireland. Hundreds of thousands of pounds have been lost in endeavouring to utilise these deposits to obtain oil and charcoal. The charcoal is valuable for metallurgical purposes, and has to some extent been employed in Sweden for the manufacture of Swedish steel. The yield of oil from the dried peat is high, owing to the large amount of volatile matter, and the oil is of a good quality, and upon fractionation gives motor-spirit, light and heavy fuel-oil, paraffin wax and pitch. For example, by the carbonisation of a Scotch peat which contained 14·6 % of moisture and 53·5 % of volatile matter I obtained for each ton retorted :—

Spirit	1·9 gallons	(the gas was not scrubbed, otherwise the yield would have been higher).
Fuel-oil	13·8 „	(Flash 184°F.).
Paraffin wax	21·0 lbs.	
Phenols (separated from fuel-oil)	1·6 gallons.	
Retort carbon	50·0 lbs.	

In order to obtain the highest yield of fuel-oil, it was found advisable to distil with superheated steam, and to continue down to carbon. The figure given for fuel-oil is after extraction of the phenols.

Beside the above products there was also obtained 2·5 galls. of wood-spirit (a mixture of methyl alcohol and acetone), 23·8 lbs. of acetate of lime, and 8·0 lbs. of sulphate of ammonia.

I will not dwell further upon peat, except to remark that when a good and economical process for drying peat on a commercial scale is devised, then peat may be one of our chief sources of oils.

At present the greatest source from which oils can be obtained is coal and cannel. The industrial supremacy of Great Britain has, in the past, been almost entirely due to her coal resources. We have been using up this capital upon which, to a large extent, the existence of the British Empire depends. We have been exporting it to other countries in enormous quantities (I am not now referring to war exigencies), and generally behaving as if there was a limitless supply that will last for countless generations. Without necessarily being a Protectionist in the old sense, I would certainly put an export tax on all coal sent abroad. At any rate this would help to pay the expenses of the war and of reconstruction. This would probably not mean a saving of coal, because if other nations require coal they will be quite willing, in fact compelled, to pay for it. If, however, instead of burning coal in open grates as we do, the coal were first subjected to treatment to remove the bulk of the volatile matter, and make it compulsory to use a smokeless fuel, then, even if there were not an actual saving in the coal mined, all the products which could be obtained from it would be utilised, and there would be no waste.

Furthermore, look at the improvement in the atmospheric conditions of our large cities and towns. No more smoke! Think of the saving to the decorations! A smokeless fuel containing from 8 to 9 % of volatile matter is readily ignited, burns with practically no flame or smoke, and gives out a greater heat than that produced by raw coal.

Coke produced by high-temperature carbonisation is not generally liked by the householder, and it is not very suitable for the ordinary domestic grate, where, unless carefully tended, it is apt to die out. The average person, therefore, will not use it unless it is mixed with coal, and most domestics use 90 % of coal, so that the saving is not very great.

If all the coal employed for domestic purposes, amounting in 1913 to between 35,000,000 tons and now about 45,000,000 tons, were to be partially carbonised, and taking the yield of crude oil at the very low rate of 20 gals. per ton (the one thing I wish not to do is to exaggerate, but many coals will give very much higher yields than 20 gals.) the production of crude oil would be 700,000,000 to 900,000,000 gals. per annum. Or putting it in tons, and taking 240 gals. to the ton, the production would be from 2,900,000 to 3,750,000 tons. Further, taking the yield of sulphate of ammonia at 10 lbs. per ton of coal carbonised, the production would be 156,000 tons per annum, and everyone knows the value of sulphate of ammonia as a fertiliser. But beside the waste of coal for domestic purposes there are other directions in which much coal is wasted. In coal mining a great deal of dust is produced, the bulk of which is left in the workings. This dust, unless kept well damped, may cause very serious explosions. The dust is generally of the same composition as the coal, and if brought to the surface could be treated by low-temperature carbonisation, and smokeless fuel and by-products produced from it. Last week I had a sample of coal sent to me, which gave the following analysis :—

Volatile Matter (on dry material)	28·47 %
Ash	21·41 %
Fixed Carbon	50·12 %

This coal coked very well, and after the volatile matter has been taken off could be gassified on a producer. The ash is rather high for the residue to form a really satisfactory domestic fuel. At present I am informed that about 700 tons of this coal is being brought to the surface and thrown on to the dump-heap. Here is a terrible waste of material and labour, and I am informed that thousands of tons of coal are mined and brought to the surface, merely to be dumped on the waste-heap. There is ample material to be dealt with, and as a large portion could be carbonised and gassified on producers for power purposes, railway tracks could be electrified, and a large portion of coal which is used for locomotive purposes saved or better employed. Although most people admire the majestic steam locomotive, yet from the point of view of utilisation of the energy in fuel, it is a very wasteful machine. In the

year 1913 over 15,000,000 tons of coal was consumed on the British railways. If all the branch and short lines were electrified, using steam traction only on the main lines, what an enormous saving of coal there would be.

Without prophesying, I think we can all look ahead and visualize the time when the energy in the coal will be utilised in this manner.

In order to get the greatest use out of coal, undoubtedly the commonsense method is to carbonise or semi-carbonise, obtain by-products from it, and produce a fuel which can either be gassified on a producer, or employed as a domestic fuel, and also for firing boilers. The question, of course, is to find and adopt the best method of retorting.

As is well known to members of this Institution, the Government were pressed to give facilities for testing various retorts to ascertain which would be the best for big-scale work. Instead of assisting those who had experience with fuel-oils, and granting facilities for erecting plant to supply the very much-needed oil, they approached the Gas Works, and asked them to produce the fuel-oil. Now, the Government were requiring at the same time benzol, toluol, etc., but it is not possible to produce fuel-oil, except in the form of creosote, at the same time as benzol and toluol. Why then did they ask the Gas Works to carry out tests by heavy steaming of cannels, and obstruct and withhold facilities for putting up low-temperature carbonising plant? As a war measure there was no objection to employing both processes, and I must say that the gas engineers rose to the occasion. Dr. Coleman, that veteran Mr. West, and also Mr. Bond, worked at a process for steaming on vertical retorts, and were able to produce a fairly good fuel-oil. But what did it amount to?

The Mineral Oil Production Department undertook to adapt the following installations of retorts:—

Nottingham	(started	May	1918)	treated	150	tons	of coal	per day.
Dundee	(„	Sept.	„)	„	200	„	„ „	„
Bradford	(„	„	„)	„	200	„	„ „	„
Bolton	(never	completed)						
Nelson	(„	„	)					

Nottingham worked 7 months, and produced 945,000 gals., Dundee and Bradford worked 2½ months, and produced 450,000 gals. each, making a total production of 1,845,000 gals. The cost of conversion of the retorts was £100,000. The average cost of material treated 25s. per ton. The average amount of distillate obtained is stated to be 30 gals. per ton of material carbonised, which makes the cost

of the oil at 1s. per gal. The coke is practically valueless, owing to the action of the superheated steam on the free carbon.

In low-temperature carbonisation, either good or poor coal can be employed; in high-temperature practice, it is always advisable to use the best.

I do not intend to go fully into the relative merits of the different low-temperature processes which have been devised, and before referring to them I might just mention other sources from which small quantities of oil are obtained.

Blast Furnaces (in which coal is used as fuel in place of coke).—A heavy tar is condensed from the gases, and from this tar heavy and light fuel-oils are obtained. The higher fractions are not sufficiently viscous to form lubricating oils. The volatile matter is driven off at comparatively low temperatures, as the upper portion of the furnace, where the coal enters, is relatively cool compared to the heat of the smelting zone. The oils appear to be intermediate between true low-temperature oils and the products obtained by the high temperature of the gas retorts. They contain sometimes as much as 25 % of tar acids, which are employed for making disinfectants. As coke is employed in the majority of blast furnaces, the quantity of fuel-oil obtainable only amounts to a few million gallons per annum.

Producer Gas Plant.—Where coal is gassified on a producer, tar is obtained and, on distillation, yields fuel-oils. This is also a low-temperature product, as the temperature at the top of a producer does not as a rule exceed 600° C., and may be lower.

The amount of tar obtained from the existing producers of 1,000 h.p. and over appears to be about 70,000 tons per annum. The tar, however, is not generally refined, and is considered rather a nuisance than otherwise. I have not examined many samples of producer tar, but those I have tested have yielded quite good fuel-oils.

Distillation of Pitch.—When pitch is heated in a retort to a temperature of about 600 to 650° C., it gives up its volatile matter, and a heavy tar condenses out. This tar on distillation gives a heavy fuel-oil. One ton of pitch yields over 20 gals. of this oil. It answers to the Admiralty tests for Navy fuel-oil, and, seeing the enormous quantities of pitch produced at the Gas Works, it might be used as a source of oil. The residue left behind is a porous carbon similar to retort carbon obtained by cracking oils, and can be used in the manufacture of electrodes. Owing to certain difficulties in constructing suitable retorts, pitch has never been worked up on any considerable scale. During this year I have

distilled about 8½ tons on specially-adapted horizontal gas retorts at Southport with satisfactory results.

Low Temperature Processes.—I have already described the shale-oil retorts in considerable detail, because it is an established industry, and it will not be possible in the scope of this paper to do more than mention some of the principal proposed low-temperature retorts.

The Chiswick Retort.—This is one of the best known retorts. It is essentially a long cast-iron tube, containing a worm conveyor of the Archimedean screw type. The shaft of the screw is hollow, and is heated by means of a Bunsen gas-flame, consequently the radial portion of the screw is also heated, and transmits its heat through the material as it is slowly carried forward. The tube, which has a length of 20 ft., and a diameter of 2 ft., is built into brick-work flues with a slight downward incline. The material to be treated is fed in by means of a sealed hopper (so that no air can enter), is carried slowly through the retort, and finally discharged into a closed receptacle. The volatile vapours, as they are produced, are drawn off and condensed. The gas is used to heat the settings, which are kept at temperatures ranging from 350° to 450° C. The objection to this retort is that it will only deal with shales, cannels and non-caking coals. If the coal swells as it is coking, the blades of the screw become blocked, and the screw ceases to work.

The Tarless-fuel Retort.—The name of this retort denotes that the retort produces a fuel which does not give off smoke when it is burned. The retort is vertical, and consists of a cast-iron tube having within it two concentric annular chambers, which are connected to the sides of the main retort by means of radiating arms, which divide the annular spaces into three chambers. These arms also act as heat carriers. The retorts are built vertically into a firebrick chamber, which is heated either by the gas given off in the carbonisation or by gas from a producer.

The retort has air-tight doors at the bottom and top, the bottom one being for discharging, and the top one for charging. The carbonisation takes place under a vacuum of 14 or 15 ins. When the charge commences to gas freely, it is rather difficult to maintain so high a vacuum, but this of course depends upon the capacity of the pump and the effectiveness of the condensing system. The carbonisation is conducted at a temperature of about 480° C. Either caking or non-caking coals can be carbonised on this retort, but there is a tendency for the coke to " hang up " with coals which swell very much on coking.

Richards-Pringle Retort.—This retort has a larger capacity per

unit than either of the two previously-mentioned retorts. It is horizontal, and consists of a firebrick chamber, through which travels an endless band, something similar to the grate employed in mechanical stokers, except that the chain has V-shaped boxes into which the coal is charged at one end, and from which the coke is discharged at the other end. The boxes open out as they pass over the end, and close again when they pass up and resume the horizontal. This method of opening out prevents the coke adhering to the sides of the boxes, which would otherwise make it difficult to discharge.

The process is continuous, and the gases and vapours are drawn off at two points on top of the retort.

Lamplough's Retort.—This retort has only been made on a small scale. Lamplough relies upon superheated steam to drive off the volatile matter.

Turner's Retort.—In this retort also super-heated steam is employed. The retort consists of a cylindrical iron container 2½ ft. in diameter and 7 ft. high. Steam at 20 lbs. pressure, which is super-heated to 500° C., is admitted into the container until a pressure of 5 lbs. per sq. in. is registered. The steam-valve is then closed, and the pressure released until 2 lbs. is indicated on the gauge. Steam is again admitted, and the alternate process repeated until no further distillation products are observed. I am inclined to think that a retort of this kind would require too much supervision, and that consequently, with a large battery of retorts, the labour-charges would be very high.

Easton Retort.—This retort is somewhat after the Chiswick pattern, except that two or more intermeshing propelling or feed-screws are employed for carrying the material forward through the retort. The inventor claims that this intermeshed screw arrangement could be employed in coke-ovens, in order to render them continuous in working. Naturally as the screws consist of steel it would not do to employ them for high-temperature operations, and consequently the ovens would require to be worked at lower temperatures to produce oil and soft coke.

Vertical Gas Retorts.—It may prove that vertical gas retorts, especially constructed for low-temperature operations, will be found to give the best results for obtaining oil from bituminous material. It will not, however, do to rely solely upon the employment of a large volume of steam, otherwise a poor coke will result. In conjunction with the Nitrogen Products & Carbide, Ltd., I have devised a vertical retort something after the Duckham and Glover-West pattern, in which steam and stripped gas are passed through

the charge, but it is intended that only the minimum amount of steam shall be employed.

The operations carried out at Nottingham and Bradford on the Glover-West retorts have proved that fuel-oil can be obtained on large vertical retorts, and I understand with a fairly good throughput. These retorts were, however, constructed for the production of gas and not oil. Probably better results could be obtained on vertical retorts specially constructed for the production of oil and smokeless fuel.

Other retorts might have been mentioned, but I think the examples given are sufficient to illustrate the lines on which work has and is being mainly carried out.

DISCUSSION.

The President expressed the indebtedness of the members to Dr. Perkin for his very interesting paper. He himself was not qualified to take part in the discussion as to the relative merits of high- versus low-temperature distillation, but he had no doubt that there were a number of members present who had views on the matter, and they would be very glad to hear them. There was, however, one point as to which he desired some explanation. The author mentioned at the beginning of the paper that the yield of sulphate of ammonia with low-temperature distillation was very much lower than with high-temperature, but later on, when he came to give some examples, he gave a very much lower yield for high- than with low-temperature distillation.

Another point that had been mentioned in the paper was the provisional suppression by the Censor of the paper read before the Institution in February last year. He did not want to go into the reasons for that course, but he thought they were all agreed that, now that happily the necessity for the censorship was over, every possible light should be thrown on to this question of high- versus low-temperature distillation in view of the enormous importance of the question from both the economic and national points of view.

Prof. J. S. S. Brame said the very interesting paper of Dr. Perkin's raised some points of great national importance. It was a very great tribute to British industry that the shale-oil industry had held its own, and developed as well as it had in spite of what might be regarded as overwhelming natural difficulties in competition with a natural product, and he thought it augured well

for hope that in this country an equally successful system of distillation would be applied to other products than shale. He was sure we should not have to turn to German chemists to tell us how to do this, or to German engineers to tell us how to design and make our plant. But there was a great number of problems which would have to be solved before we should come to the successful production in any large quantities of oil suitable for our general purposes, oil which could be compared with natural petroleum oils. Dr. Perkin had indicated some of the directions in which we required further knowledge. A great deal had been done in recent years on distillation of coal at low and high temperatures, but nevertheless the results were often very contradictory, and we were largely in the dark as to some of the reactions. That probably arose from the fact that coal itself was such a very variable substance, which might account for the different results obtained in this country and in the United States. It was exceedingly difficult with a problem like destructive distillation to differentiate between primary actions and secondary reactions. We had in the first place the evolution of certain products from the coal as the coal was heated, and in the second place we had a breaking-down, a complete transformation in some cases, of these primary products at higher temperatures, either in contact with the walls of the retort, or in contact with the heated carbon of the retort, and these were problems which of course were very difficult to solve.

There were one or two interesting processes which had not been mentioned in the paper, and to which he would like to refer. Probably some of them were familiar with the process that was devised some time ago by Mr. Maclaurin of Glasgow, in which he passed highly-heated producer-gas through his carbonising retort, and in that way obtained some oils which were very different from anything else that had been described in the literature of oils. These oils were almost wholly soluble in caustic soda, and that brought him to an interesting point in connection with cannel oils and other oils from coal, viz., the oxygen-containing compounds. Many of these low temperature tars yielded an enormous amount of soluble phenols and higher phenols, and it was not unusual to find in treating the fractions that probably 30 % was soluble in caustic soda, but that did not remove all the oxygen-containing substances. They had in addition, as Windmill had shown in 1917 (*J. Soc. Chem. Ind.*, xxxvi, 912), in a paper on the distillation of cannel coals *in vacuo*, phenolic ethers, which were not removed by caustic soda, but which could be removed by strong sulphuric acid. Windmill showed that the average loss of his distillates

to caustic soda was 35 %, and that in the 200° to 220° fraction it was 47 %. Of course these phenolic ethers might or might not be substances which would have to be removed from the oils in order to give products which were suitable for certain purposes, such as use in internal-combustion engines. It was well known that certain oils gave a great deal of trouble in such engines by carbonisation, pitting, etc., and no one could tell at the present time the cause of these troubles. Very little apparently was known of the effect of these oxygenated compounds. He believed they had a large influence on the behaviour of these oils in engines of the Diesel type, and the preparation of fuel-oils for use in internal-combustion engines would undoubtedly become one of the most important phases of this new industry.

In another process, recently developed in America, it was proposed first of all to carbonise the coal at a temperature of 450°, and in that way get off as large a quantity as possible of the low-temperature oils; then that the residual coke should be briquetted with pitch, and these briquetted masses again retorted, to recover a further quantity of heavy oils. It was claimed that no matter what the origin of the material might be, the final residue made a good domestic fuel (*Journ. Soc. Chem. Ind.*, xxxvii, 679A. 1918).

Dr. Dunstan said there were so many points raised in the paper that it was difficult to select a few for discussion at the moment. He was, however, particularly pleased with Dr. Perkin's crusade *à propos* of the claims which had been made in connection with the formation of benzol and toluol in low-temperature processes. It was perfectly obvious, of course, that a temperature of 700° C. was about the minimum for the formation of these bodies. At the same time there was a considerable amount of misapprehension as to the production of those valuable aromatic hydrocarbons. In common parlance coal tar was referred to as the source of these materials, but in point of fact it was the stripping of coal-gas which yielded them in quantity. Before the war the great bulk of the coal-tar made in this country went on to the roads.

In connection with the paper as it stood, he would like to emphasise the extreme importance of a thorough research on the composition of coal and of different types of coal. He would like to remind the members of the admirable monograph by Dr. Wheeler and Miss Stopes on the constitution of coal, recently published by His Majesty's Stationery Office. That brought the subject well up to date, but the extraordinary discrepancies between the results, for example, of Burgess and Wheeler on the one hand, and Pictet and Bouvier on the other, showed quite clearly that the material

with which these investigators worked was different in type. He might perhaps mention one point in particular. Jones and Wheeler discovered that the cellulosic component of coal yielded phenols, whereas Pictet and Bouvier found that no phenols at all were obtained. It was gratifying in this connection to realise that all the pioneer work on the composition of coal had been essentially British. The first investigations by Bedson who used pyridine as solvent were classical, and all subsequent investigators had followed the lines he laid down, and had shown that a coal substance, generally speaking, could be divided into two classes, cellulosic and resinic. The cellulosic was that part which provided phenolic components on distillation, and the resinic part was that portion which provided hydrocarbons. Pictet and Bouvier discovered from the solubility of coal substances in boiling benzol that they obtained a series of hydrocarbons identical with those occurring in petroleum, and they made the somewhat striking assertion that coal was a solid hydrocarbon impregnated with liquids allied to petroleum. When one remembered that coal was usually described as a form of carbon, it was striking to realise that possibly coal was a complicated hydro-carbon itself. The crux of the whole position of low- and high-temperature distillation was concerned with the curious fact that at about 700° there was a marked change in the nature of the products. Below that temperature essentially a mineral oil was obtained, whilst above that temperature a large evolution of hydrogen took place, and the well-known aromatic hydrocarbons were produced.

It was possible to deal at great length with the different points in connection with low-temperature tar, but at that hour of the evening he would pass on to a discussion of the work of Parr, a chemist of Illinois, who three or four years ago subjected a number of American coals to low-temperature distillation, and *à propos* of Prof. Brame's remark, he also found the tar acids comprised about 30 % of the total oil. Parr stated that the semi-coke that he prepared, which had 18 % of volatile matter, worked well in a suction gas-producer, and was equal, or nearly equal, to anthracite. The volatile content rendered it admirable as a domestic fuel; it was clean, dustless, smokeless, and burned well and evenly. The tars produced had 1·07 gravity, were rich in oils (18 % at 200° C.), and had a low content of pitch, which in turn was particularly low in free carbon. The tar acid, Parr pointed out, was admirable from the point of view of a wood preservative, as a natural creosote. The 10 % distillate of the low-temperature oil, which was free from acids, could be used directly in internal combustion engines. The

point which struck him very forcibly about Parr's paper was that he described that these low-temperature oils as distinct from coal-tar oils, and as offering a new field of research altogether.

He noticed that Dr. Perkin rather suggested that these oils were of no use for synthetic purposes, but he hoped that he would prove to be very much mistaken in this view, and that in the low-temperature carbonisation process we should find bodies which would be of extreme importance in synthetic chemistry.

He would raise one point in connection with ammonia because that was highly important when considering the technology of the low-temperature process. It had been shown by Cobb that at 800° C., in the presence of steam, it was possible to obtain practically the whole content of nitrogen as ammonia. In any process which aimed at obtaining the maximum yield of ammonia, this fact must be borne in mind.

Sir Boverton Redwood wished to emphasise the fact that since this subject was previously under consideration by the Institution, circumstances had happily been completely altered by the signing of the armistice, and they were now in a position to do what they could not do last February, viz., to lay the foundations of a permanent industry on a post-war basis. In connection with that he felt very strongly that there ought to be carried out, as soon as possible, investigations with the object of placing the true low-temperature system of distillation on the same basis in the matter of practical commercial experience, so far as might be possible within a year or two, as that on which the high-temperature system of distillation as practised in the Scottish shale-oil refineries existed at present. He confessed that he was a little surprised to hear Dr. Perkin—whose qualifications to express *ex cathedra* opinions he did not in the slightest degree challenge—refer to the Scottish practice in relation to obtaining oil, as one of low-temperature distillation. No doubt theoretically it was a process of low-temperature distillation in the sense that the Scottish practice aimed at taking off the volatile oil products at the lowest temperature at which they could be effectively and economically separated, but when one took into consideration the diameter of the Scottish type of retort, it must be perfectly evident that in order that the centre of the charge should be raised to a temperature sufficiently high to effect that separation, even at the lowest available temperature, the exterior portion of the charge in contact with the walls of the retort must of necessity become overheated, and that occurred long before the shale travelled down from the upper portion of the retort, in which the oil was supposed to be separated,

into the lower much more highly heated section where the ammonia was obtained. That the result was relatively unsatisfactory from the oil point of view was, he ventured to submit, definitely shown by comparing the yield of oil from a cannel coal in quantity and quality as obtained in a true low-temperature system of distillation, with that obtained in one of the Scottish shale-oil works. It was quite evident from a comparison made in that way that there was overheating of the charge, and he submitted that what was wanted at the present time more than anything else was practical testimony based upon large-scale commercial test with true low-temperature distillation of materials which, as Dr. Perkin had shown, were at the present time largely unutilised and might be made a source of great quantities of oil which was so badly needed.

He had only to say in addition that, as would be remembered, the outcome of the discussion of this subject last February was the formation by the Institution of what he could, without exaggeration, characterise as an exceptionally influential and representative Committee, to consider this matter, the terms of reference being: "To obtain evidence in respect of the quantity of cannel coal and allied minerals available in Great Britain as a source of motor-spirit fuel-oil, and other products, and to formulate a scheme for the utilisation of such supplies." As to the activities of the Committee, they had not full official information, but it was known to a large extent what they had done between the date of their appointment in February last and the month of July, when an interim report was made public. In that interim report there was the statement that the Committee had already as the result of their investigations obtained evidence which they held to justify them in making the statement that at least 10,000 tons a day of retortable material could be economically assembled for treatment, provided the necessary facilities were available. That report was made last July, and the references to Government facilities and labour were made under war conditions, but, as he said at the commencement of his remarks, he felt that it was very desirable clearly to recognise that the disabilities which at that time existed in relation to the utilisation of this large quantity of waste material, did not any longer exist; at any rate, they were in process of being rapidly removed. The necessary labour was coming back, and the necessary materials for the construction of plant could certainly be obtained with less difficulty than was the case previously. We were further told in this interim report that, as the result of tests carried out, it had been ascertained that a yield of between 15 and 80 gals. of crude oil per ton could be obtained from existing

materials in the British Isles, which would give 300,000 gals. of crude oil a day or upward of 400,000 tons a year. In one respect, this report had already borne fruit, and the importance of having practical men on that Committee was shown by the fact that attention had been drawn to the very important result which would accrue from the simple change of substituting the shovel for the fork in the mining of these bituminous materials, the effect of the fork being that a large amount of material available for this particular purpose was left underground. As he had said, in one respect the report had borne fruit because he had had his attention drawn to a public announcement that negotiations with the owners having been fruitless, a ballot of the Leicestershire miners had shown a majority considerably over the necessary two-thirds figure in favour of a strike to enforce the filling of coal with shovels instead of forks, which had been a vexed question for some years. Having regard to the importance of such a source of oil there ought surely to be no difficulty in making arrangements for the utilisation of this material on a large scale, and those who had had the advantage of listening to the lecturer would go away feeling satisfied that if any scheme of this kind was carried out it would not be necessary to go abroad for professional help in giving effect to it.

Dr. Ormandy remarked that the paper offered so many points for discussion that he hardly knew which to choose. Dr. Perkin had emphasised the great importance of the question of the value of the sulphate of ammonia as a factor in the development of the shale-oil industry, and he was afraid that in the future we should have to consider these commercial factors much more than we had done in the past. We must realise, for instance, that whereas up to now we had always been about the largest exporters of sulphate of ammonia, we were now faced with the fact that America was a large producer. China was starting to produce, being now putting up by-product coke ovens. Japan was a large producer, and Germany was stated to be making more than one million tons of sulphate of ammonia from the nitrogen of the atmosphere. That looked as if we were going to be up against a very serious situation as regards the value of ammonia compounds, although we in this country had not in the past used ammonia fertilisers as we should have done; our consumption was only a fraction of that of Belgium and France. We were therefore up against the fact that the material was going to be cheaper in the future than it had been in the past, and he wished to emphasise the fact that what had been controlling factors hitherto would not be so in the future.

With regard to lignite, it had been distilled in very large

quantities, particularly in Austria, for many years. He once visited some very large works outside Teplitz where there were about 70 by-product recovery coke ovens into which the brown coal, which was dug in open workings, taken out by a grab, put into wagons, and run down an incline, was dropped straight into the retorts at a cost of a few pence per ton. The residue left from the distillation in the ordinary by-product recovery coke oven was ground, and the pitch and tar produced from the distillation was used to briquette the carbonised lignite. These briquettes were largely used on the State Railways of Austria. They also had a unique system of dealing with the gases coming away from the coke ovens. These were led through mains jacketted with magnesia, as also was the main pipe going to the washers, and the gases containing tars and oils were cooled fractionally, and washed in vertical rotary washers, so that in the first washer the heavy tar was condensed, in the second, light tar, in the third, heavy oil, and from the fourth washer there was constantly coming out a stream of pale yellow oil, which was sold locally, in competition with Russian and other petroleums, for lighting without any further refining whatever. It was a utilisation of the Feld principle, which he thought would have to be adopted some time or other, instead of the stupid arrangement which obtained to-day, whereby we distilled all the various products, condensing them down into one sticky mass, and then proceeded to heat them up again, causing a large number of secondary changes, and at great and needless increase of cost. It was an unscientific method at best.

With regard to lignites on the South Coast, Nature seemed to have placed them there intentionally. It was the most distant part of the country from the coalfields, but if we could get cheap fuel there, he saw no reason why the work of the congested Potteries should not be transplanted down to the more beautiful air of Dorsetshire and Devonshire, and the industry developed on new lines, where it would be possible to employ lignites profitably. The advantageous utilisation of lignite in its early stages might be problematical, because the lignites near the surface were comparatively high in ash, but as they went deeper and deeper the quality got better and better, and beds 30 ft. to 40 ft. thick had been tested by boring; the lignite was there very much freer from ash than it was at the top. From these lignites it should be possible to obtain a considerable quantity of Montan wax, which, as Dr. Perkin had said, was not found in rich seams of wax-bearing lignite, but in comparatively isolated pockets, and as the coal was brought up, the wax-containing material was simply hand-picked and sent

down to the extractors, and in this way from 10 to 40 % of wax could be obtained. If they could get cheap power, it would pay to work these lower beds of lignite, because in order to reach them they passed through 300 or 400 ft. of clays of all degrees of purity, many of them of the highest quality, not hitherto used for the pottery trades because they were mixed with some fine sand which rendered them not plastic enough for the purpose for which potters required them. But if cheap power was available, the electrical method for the purification of clay could be used, and there was no reason why a valuable industry should not be developed down there. The reason that the pottery industry had gone to Stoke and the neighbourhood, was not because the clays were there, they being obtained largely in the South of England, but because the coalfields were near. It required about five tons of coal to burn one ton of pottery, and if cheap fuel was available on the South Coast, the potteries could be placed on the spot where there was a high percentage of the clays now used in the North of England. At present it was complained that the potteries could not be modernised in many cases, because of the lack of space. The existing works were old and horribly congested, but in the better surroundings of the South of England the long tunnel-oven and other improvements could be introduced. If these clay beds were properly developed, and the best method of working them was fully investigated in the laboratory, there was no reason why the South of England, which hitherto had failed to be a manufacturing district, should not become a great source of wealth.

On the motion of **The President**, a hearty vote of thanks was accorded Dr. Perkin at the conclusion of the discussion, to which there was not time for him to reply.

Subsequent communications were received as follows:—

(1) From **W. H. Manfield.**

I quite agree with what Dr. Perkin has said with regard to the quantity and quality of the products obtained by the carbonisation of bituminous minerals varying greatly according to the method of retorting employed and upon the temperature at which the process is conducted. This is especially the case when dealing with oil shales. I have found that a slight alteration in the temperature at a certain point of the distillation has increased the yield of oil by 15 %. In some retorts a portion of the oil-vapour is condensed by the cold charge coming into the retort, carried into the warmer portions of the retort, and volatilised again, causing a variation in the yield of the products. In other retorts the volatile matter

set free at a low temperature is not expelled before becoming changed by further heating.

Although perfection in retorting has by no means been yet reached, the great amount of research work that has been done has ascertained the methods of treatment by which very good yields of oil and other products can be obtained from different bituminous minerals, and I am of opinion that some little incentive such as the permitting of the sale of home-produced motor-spirit free of duty, or the granting of a subsidy equivalent to this, would soon lead to a very large industry springing up, giving employment (which will before long be so much needed) to a very large number of men. I recommend that the Government should be urged to give the Home oil industry some such encouragement.

(2) From **Mr. A. B. Thompson.**

Dr. Perkin has in his lecture emphasised in an attractive way indisputable facts concerning a comparatively new enterprise which will one day acquire enormous importance. It is unfortunate that, whilst the chemical features have been widely discussed and generally agreed upon in principle, the equally important mechanical aspect has been seriously neglected, mainly because practical demonstration on a commercial scale entails heavy expenditure. Shales and lignites necessarily vary so much in composition that no standardised form of plant is applicable to different deposits. Failure to appreciate this point has been one of the main causes of the non-success hitherto of all shale enterprises outside Scotland, where long experience has developed a process applicable to those particular deposits. The attainment of laboratory results on a commercial scale is very difficult in practice, and most of the experimental plants erected for bulk tests can only be regarded as a rough guide to what may be expected in larger plants. Brief runs of a few days' duration, which usually characterise test plants, rarely expose the mechanical difficulties that a continuously running plant develops. My contentions are well illustrated by the slow development of efficient cracking plants, although the essential chemical facts were known very many years ago, and the rewards of success very great. Experimentalists still expect impossibilities from steel and iron in the design of plants which last but a few weeks in continuous practice.

That the distillation of oil shales and carbonaceous minerals will one day be undertaken on a vast scale is a foregone conclusion, but whilst ready-made oil can be produced so cheaply from oilfields, there is a doubt in my mind as to whether the distillation of shales, etc., can be profitably undertaken in competition with natural oils,

except in a few highly favoured localities, especially as the labour involved in mining is likely to reach such a high figure in the near future. Certainly the profits can never approach those of successful oilfield development where the returns on capital expended in drilling are often fabulous.

The lecturer's reference to exaggerated claims touches one of the disturbing features of past efforts. On several occasions in examining shale propositions where remarkable potentialities were described and supported by authentic but incomplete and dangerous data, millions of tons of shale had been calculated from restricted outcropping seams which were rarely homogeneous, and often contained numerous partings of valueless or low-grade material for which no allowance had been made. Not only were the chemical results based on selected bands in a mixed body, but no thought was given to the costly sorting that would have been necessary for retorting. Even assuming fine-bedded sediments like oil shales to be more continuous than oil sands, it is certainly inadvisable to assume almost indefinite prolongation of seams in the direction of mining without very exact data from borings. Complex mining problems are also introduced in the excavation of inclined and sometimes disturbed seams, especially where uncertain hanging walls exist. Calculations of available tonnage need a very good judgment.

Supposing, however, that all the essentials for satisfactory extraction and treatment exist, one still wonders whether distillation methods can compete with natural oils until the great oilfields of the world are much further exploited. More materials and labour are needed for the extraction by distillation of oil from mineral substances than in the development of oilfields; consequently, increasing costs will fall most heavily on this potential industry. Against this might be offset in the case of British deposits some advantage of labour market and freight cost over oils drawn from distant countries where natural oils exist.

If the American figures of oilfield losses and waste are no exaggeration, the attention of economists could more profitably be applied to the diminution of the appalling losses of oil sustained under present systems of working.

A recent investigation by the Washington Bureau of Mines led to estimates, substantiated by incontrovertible figures, that should compel oilfield operators to reconsider their methods. All those associated with oilfield development know that oilfield waste continues on new oilfields nearly as much as formerly, and that all the platitudes about prevention of oilfield waste have availed

nothing outside a few of the American fields where the fabulous losses aroused public resentment. Tens of millions of cubic ft. of gas still pass daily into the atmosphere unused and irrecoverable, carrying yearly with it millions of gallons of extractable gasoline valued at hundreds of thousands of pounds sterling. The comparatively small profits derived from the utilisation of the gas are so insignificant compared with the huge profits resulting from the main product of search in new oilfields, that capitalists will never be induced unaided to expend their resources on such low-yielding enterprises. Nevertheless in many cases much greater profits would result from the treatment of such wasted products than in the distillation of carbonaceous shales.

The distance of many oilfields from centres of industry which could utilise gas or electric energy diminishes the uses to which gas could be utilised, but the extraction of gasoline, long-distance transmission of gas, the fixation of nitrogen from the air and the development of local industries represent some of the ways in which Governments could assist in reducing the present waste of natural resources.

Dr. Perkin's reference to the amount of water that tars will hold without being apparent I can confirm. In some liquid-fuel tests with water-gas tar some years ago, surprise was felt at the low heating value in the furnace compared with previous results. On testing the oil it was found to contain 50 % of emulsified water.

Dr. Perkin replies as follows :—

In reply to the President the lower yield of sulphate of ammonia referred to the actual retorting of the coal or cannel. The higher yield is that obtained when the residual coke, containing about 8 to 10 % of volatile matter, is put through a producer, as against that obtained from hard coke, containing very little volatile matter.

Prof. Brame has dotted my i's and crossed my t's in his interesting remarks. Dr. Dunstan said I rather suggested that the oils had no use for synthetic purposes. In our present state of knowledge I am afraid that is so, but who knows but what further research may bring forth. No one knows better than Dr. Dunstan the extreme difficulty of dealing with these oils and purifying them. That of course should not make us despair of obtaining valuable products which could be used for synthesis. Take coal tar, for example, from which many valuable products are obtained, these products only form a comparatively small portion of the tar, and we have a great deal to learn as to the constitution of the residues.

I quite agree with Sir Boverton Redwood that lower yields of

oil are obtained from cannel when it is carbonised in a Scottish shale-retort, and I have always considered that these retorts are not so suitable for carbonising coal or cannel, as is for example the Chiswick or tarless fuel retort. I do not, however, quite agree with Sir Boverton Redwood's explanation. The temperature of the walls of the retort depends upon the temperature of the surrounding flues. If these were very highly heated there would be breaking up of the oils and carbon would be deposited on the walls, just as in the case of the highly heated walls of a gas-works vertical retort. Owing to the "scurf" as it is called, the retorts have to be periodically emptied for "scurfing," i.e. air is allowed to pass up to burn off the deposited carbon. This is not necessary in shale retorts, and I do not think the low yields of oil are due to decomposition; this could, however, be determined by the amount of gas produced from a given sample of cannel if it were retorted in shale retorts or one of the other retorts mentioned—I do not know whether such data are available.

I am glad that Sir Boverton Redwood has drawn attention to the interim report of the Committee appointed on last February, as it shows the value of the work of our Institution.

Dr. Ormandy's remarks in reference to the large amount of fuel required in the potteries is very pertinent, and bears out what I have always considered would be a great saving, that is, if gas firing from producers were employed in the potteries instead of coal firing.

With the remarks of Mr. Manfield and Mr. Beeby Thompson I am in agreement, and they do not call for reply. It is gratifying, however, that the paper should have drawn such an interesting discussion.

Thirty-Fifth General Meeting.

This Meeting was held at the House of the Royal Society of Arts, on Tuesday, 21st January, 1919, Mr. Charles Greenway (President) occupying the Chair.

The Members of Council present were Mr. Andrew Campbell, Dr. A. E. Dunstan, Mr. Arthur W. Eastlake, Dr. W. R. Ormandy, Mr. T. C. Palmer, and Sir Boverton Redwood, Bart. A message of regret for unavoidable absence was received from Dr. F. Mollwo Perkin.

The President, in calling upon Mr. A. Campbell to read his paper upon the subject of Paraffin Wax and its Manufacture, said that no one in the British Empire had a wider experience or was

better qualified to speak on the subject than Mr. Campbell, and he was sure the paper would prove to be of very great interest.

The following paper was then read, illustrated by numerous lantern-slides:—

Paraffin Wax and its Manufacture.

By A. Campbell, F.I.C., *Member*, and W. J. Wilson, A.I.C.

The term paraffin wax or paraffin is applied to the crystalline substances, solid at normal temperatures, obtainable by the filtration of distillates from petroleum, shale, lignite, etc. This definition excludes the semi-solid hydrocarbons, petroleum jelly, petrolatum or vaseline. It also excludes ceresin, the purified product of the naturally-occurring mixture of solid hydrocarbons, ozokerite. The composition of this substance has not been definitely established, but differing fundamentally as it does from paraffin wax it does not fall within the scope of this paper.

Paraffin consists entirely of saturated hydrocarbons of the C_nH_{2n+2} series ranging from about $C_{17}H_{36}$ upwards, most of which as far as $C_{35}H_{72}$ have been isolated from petroleum. The melting-points of these compounds vary from 50° F. to 169° F. The lower members of the series, liquid or gaseous at ordinary temperatures, occur also in petroleum and in natural gas.

Mabery[1] obtained the following members of the series from commercial paraffin, Pennsylvanian crude oil and deposits in wells:

	Melting-point.	
	°C.	°F.
$C_{17}H_{36}$	10	50
$C_{18}H_{38}$	20	68
$C_{19}H_{40}$	33–34	91–93
$C_{21}H_{44}$	—	—
$C_{23}H_{48}$	44	111
$C_{23}H_{48}$	45 or 48	113–118
$C_{24}H_{50}$	48 or 50–51	118 or 122–124
$C_{25}H_{52}$	53–54	127–129
$C_{26}H_{54}$	55–56 or 58	131–133 or 136–137
$C_{28}H_{58}$	60	140
$C_{30}H_{62}$	62–63	143–146
$C_{31}H_{64}$	66	151
$C_{32}H_{66}$	67–68	152–155
$C_{34}H_{70}$	71–72	160–162
$C_{35}H_{72}$	76	169

Paraffin was so named by Reichenbach[2], who isolated it from tar from the distillation of red beech, in 1830, and afterwards from other tars. Previously to this, however, Fuchs, in 1809,

is said to have observed a substance, probably paraffin, in Bavarian petroleum, and in 1819 Büchner obtained a substance from this oil which he termed "mineral fat," and which Kobell[3] in 1835 showed to be identical with Reichenbach's product. Speaking on this subject in 1874 before the Society of Arts, Field[4] stated that he considered the term paraffin a misnomer, because, in place of possessing "little affinity" it was violently acted on by sulphuric acid to which a few crystals of permanganate of potassium had been added.

Unaware of Reichenbach's discovery, the year later, Dr. Christison[5], the professor of Materia Medica at the Edinburgh University, read a paper before the Royal Society of the Scottish Metropolis on the analysis of a sample of Rangoon crude oil, found by digging a hole "near Rangoon." No doubt the source of this oil was one of the native dug wells at Yenangyoung, 275 miles from Rangoon, on the banks of the Irawadi River. Its sp. gr. was ·880 at 60° F., it had the consistency of thin paste at 86° F., and melted at 90° F., these properties being characteristic of oil from dug wells of quite recent years. Christison distilled the oil, and found a portion of the distillate to contain a crystalline body which was new to him. He thus described the treatment of the fraction known later in the industry as heavy oil and paraffin:—"The mass being cooled to about 40° F. was spread out on a filter paper. In this manner an oily-like matter was taken up by the paper, and a pale yellowish white crystalline substance was left, which was subsequently deprived of its remaining colour by repeated solution in boiling ether, and recrystallisation. Ether dissolved it largely, forming a pale yellow solution, which on being cooled by immersing the vessel in very cold water, became a soft mass of interwoven crystals. The mass was taken out, spread quickly on filtering paper, and immediately subjected to strong pressure between folds of blotting paper. The yellow colouring matter, which all remained in solution in the ether after it cooled, was thus, in a great measure, imbibed by the paper, and the crystalline matter was procured in a state of purity by repeating the process twice." The "fusion" point of the substance so recovered was 135° F. and its sp. gr. ·909 at 60° F. Christison named the new product "petroline," but later, having observed a report of Reichenbach's discovery of 1830 in Büchner's Repertorium, decided to carry his investigation no further. In 1834, addressing the same learned Society, Gregory[6] stated that from a specimen of Persian naphtha he obtained by distillation and redistillation a distillate which when exposed to cold became semi-solid, and likened its appearance

to the latter portions of Rangoon petroleum distillates obtained by Christison.

Warren de la Rue and H. Müller[7] distilled several tons of Rangoon oil with steam, wet and superheated, obtaining 96 % distillates, and by freezing and pressing the later fractions, extracted 10–11 % paraffin, which was purified with sulphuric acid, alkali and water. When redistilled, the paraffin was white, but not free from oil.

It was some years later that the manufacture of paraffin advanced to a commercial scale, and the credit of this is due to James Young[4], the founder of the Scottish, and in fact the world's, mineral oil industry[8]. To that gentleman a patent[9] was granted in 1850 "to obtain paraffine and oil containing paraffine" from shale-oil.

It appears that paraffin was first made extensively by William Brown & Co., of Glasgow, and it is on record that their wax was used by a London firm in the year 1855[4]. In 1847, Lyon Playfair[4] found paraffin crystallising in the distillate from an oil found in a coal mine in Derbyshire, a district on which interest is focussed to-day, for it is there that borings are being made for petroleum under the management of Lord Cowdray, one of the vice-presidents of this Institution. In 1849 Reece[10] obtained a patent for distilling paraffin from Irish peat—the yield in actual practice only amounted, however, to 125 lbs. per 100,000 lbs. of crude material, and the project was abandoned.

Price's Candle Co., Ltd., worked Burma crude oil in this country for paraffin wax, lubricants, and illuminants in the early 'fifties, and actually shipped some of these products to America in the years prior to the discovery of petroleum in that country.[8]

Since that period the petroleum industry of the world has developed by leaps and bounds, and the importance of paraffin wax as an asset is apparent when the world's production at the present time is considered. It is impossible to ascertain accurate figures for this, and we state 350,000 tons as a rough approximation of the quantity produced annually by the distillation of crude petroleum and shale-oil. Assuming this to be correct, and taking the value of paraffin at an average of 3d. per lb., a reasonable enough assumption, the total value amounts to nearly £3,250,000.

The chief use to which paraffin wax is put is the manufacture of candles, night-lights, tapers and vestas. The softer grades are used for dipping the splints of wooden matches. It is in demand as a water-proofing material for paper, cloth and leather, and for electrical insulation. It is found as an ingredient of floor and boot polishes and as a rust preventive. The sealing of medicine bottles and preserve jars may be mentioned among the

many other uses for which it is employed. There is probably no other petroleum product put to such a variety of uses as that we are now considering.

It remains to be seen, however, in view of the fact that the bulk of paraffin wax produced is at present consumed in the form of candles, whether the production will increase in response to a demand arising in directions as yet unforeseen, and in the face of the competition of oil, gas and electric lighting. Should it do so, the demand is certain to be met, for many of the oils rich in this substance are not worked to produce their greatest possible output.

The percentage of solid paraffin in American petroleum is quoted as 2–3 %, presumably the content only of paraffin-yielding crude oils, and not an average figure for all petroleums worked on the American continent. Scotch shale-oil contains about 11 %, Galician petroleum 5–6 % and that of Burma and Assam 12 % or more. Borneo crude oil yields an important percentage of wax, and another paraffin-yielding crude oil is that found in Persia, although as yet it has not been worked for this product on a commercial scale.

The methods in general use for the determination of the amount of solid paraffin in crude oils and distillates are too familiar to require more than passing notice. For practical working it is necessary to estimate the possible yields at given temperatures, and the most satisfactory means of doing so is a small-scale reproduction of the process to be used in the refinery.

Paraffin does not assume the crystalline form which ensures its easy separation from the oil with which it is associated until distillation takes place. It usually comes over in the distillates after the kerosene fraction has been taken off, and as the end of the distillation is reached, the higher the melting-point becomes. Take the crude from the Yenangyoung field of Burma, for instance, showing 57½ % boiling under 300° C.; the solid paraffin becomes evident in the fraction between 50 % and 55 %, increasing in melting-point towards the end, as shown by the following tests :—

Heavy oil and paraffin fractions of crude oil from Yenangyoung, Burma, distilled in current of steam under partial vacuum.

Per cent.	50–55	55–60	60–65	65–70	70–75	75–80	80–85	85–90	90–95
Specific gravity ..	0·857	0·8605	0·865	0·870	0·875	0·880	0·8835	0·891	0·909
Setting-point °F.	41*	51*	51†	71†	82‡	98‡	109‡	118‡	124‡
Flash-point °F.	222	240	276	284	302	330	334	334§	296§
Oil % on crude oil —	4·94	4·43	3·95	3·67	3·06	2·81	2·66	2·97	
Wax ,, ,, ,, ,, —	0·06	0·43	0·93	1·54	1·94	2·33	2·27	2·03	
Wax ,, ,, fraction —	1·2	8·8	18·2	29·5	38·8	45·3	46·1	40·6	
M.p. of wax °F. —	—	109	110	114	123	128	135	141	

* Scotch method. † Flow test. ‡ Stirring method. § "Cracking."

When asphaltic matter is present in the crude material, it is impossible to effect its complete separation in one distillation, for traces of it or of its decomposition-products are carried over with the high-boiling distillates, preventing good crystallisation of the paraffin from taking place. In this case it is necessary to treat a portion of the distillate with sulphuric acid, and to redistil it.

Although the sweating process is the established method for refining paraffin, information as to the actual effect of its operation to be found in petroleum literature is very scant.

The fractionation of a scale from a "well-known" shale was published by Tervet[11] as under:—

Total scale fractionated into 5 % fractions.

Fraction.	Melting-pt. °F.	Fraction.	Melting-pt. °F.
1	93	11	114·5
2	95	12	116·5
3	97	13	118·0
4	99	14	120·5
5	100	15	123·0
6	103	16	123·5
7	105·5	17	125·0
8	108·0	18	126·5
9	110·5	19	127·0
10	112·5	20	128·0

Scale is generally separated into fractions, yielding products of differing melting points. Tervet therefore gave the fractionation of the above scale when separated in two fractions:—

Fraction.	Hard. Melting-point °F.	Soft.	Fraction.	Hard. Melting-pt °F.	Soft.
1 ...	104	80·0	11 ...	120·0	99·0
2 ...	106	83·0	12 ...	120·5	101·5
3 ...	108	86·0	13 ...	121·0	103·0
4 ...	110	88·8	14 ...	122·0	105·0
5 ...	112	89·0	15 ...	122·5	107·0
6 ...	112·5	91·0	16 ...	123·5	109·5
7 ...	114	93·0	17 ...	125·0	112·0
8 ...	116·5	95·0	18 ...	127·0	114·0
9 ...	117·5	96·0	19 ...	129·0	116·0
10 ...	119·0	97·5	20 ...	130·0	118·0

As the fractionation of paraffin without having regard to the temperature and period of sweating and to the colour of the resulting wax does not convey very much information, and was only tabulated by Tervet in connection with a description of plant which

he had invented (but which was not generally adopted), we have carried out some experiments, the results of which we now place before you. The objects of the investigation were:—

(*a*) To observe the effect of variation of time and rate of temperature-increase of sweating;

(*b*) To ascertain the advantage or disadvantage of combining the scales obtained by separate filtrations at different temperatures, as compared with the separate sweating of the crude products.

The scale experimented with was extracted from the heavy oil and paraffin distillates in two stages, the hard scale melting at 129° F., and the soft scale, extracted at 32° F., at 113° F. Both were well pressed, and contained only a very small percentage of oil. When mixed together in the proportions in which they were obtained, the average melting-point of the mixture was 120·5° F.

The finished products aimed at were paraffins of 135° and 125° melting-point respectively, both to be sufficiently refined for the final treatment of filtration through decolorising earth, and to yield a perfectly white product. Generally, the melting-points of the fractions obtained varied from these standards, but for the purpose of comparison in compiling the results tabulated below the quantities obtained have been adjusted by calculation.

The scales in each case were melted in a shallow tray of suitable size, and allowed to cool slowly to atmospheric temperature. The cake of scale was transferred to a tray of interlaced wire, the bottom of which was covered by a fine gauze. The wire tray was placed in a sloping position in a larger metal tray in which the sweatings were received. The whole was contained in a large water-oven fitted with a thermo-regulator, by means of which the temperature was maintained within about 1° F. of the temperature required at each stage of the operations. At the end of each stage, the sweatings were removed and weighed, and their melting-point determined. The quantity of scale dealt with in each experiment was 500–600 grams. The diagrams A, B, C and D indicate the products actually obtained, and also the adjustments necessary to give the exact melting-points required.

Reference to the diagrams will show that in experiment A, when the hard and soft scales were sweated separately, only 17·26 % from the latter reached the proper colour, and that 58·33 % was still yellow, and required re-sweating to obtain the maximum yield of white wax.

Experiment B, in which the scales were combined proportionately, yielded no less than 41·8 % white in one operation, and

DIAGRAM A.

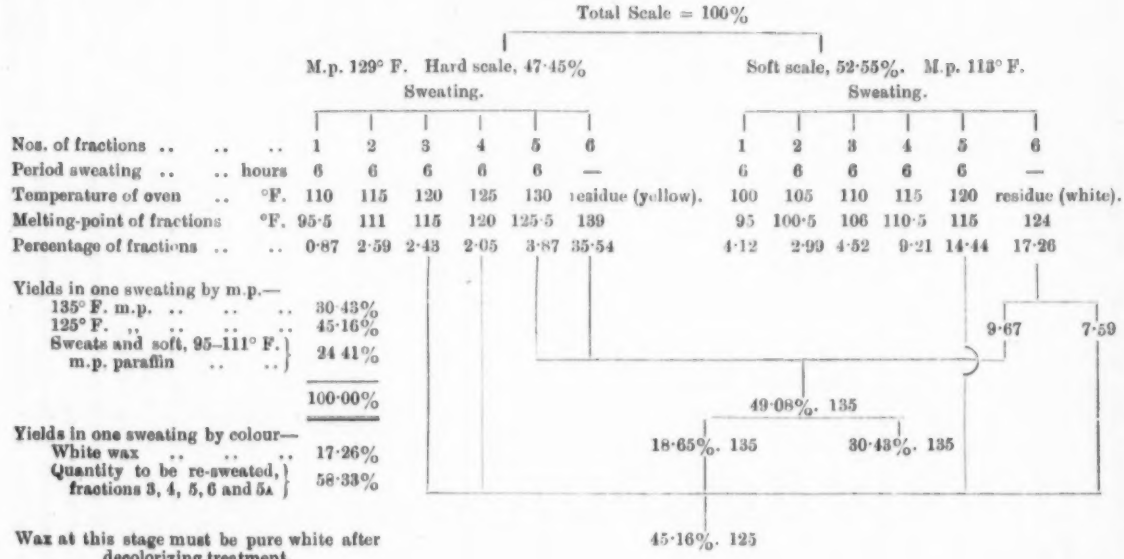
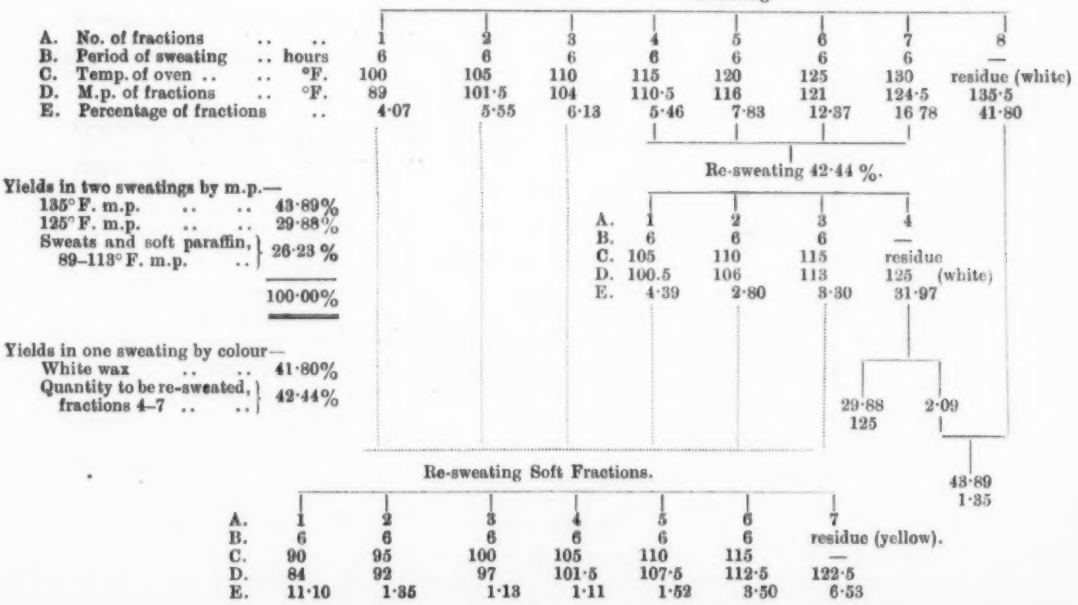

Total Scale = 100%
M.p. 129° F. Hard scale, 47·45%
Sweating.
Soft scale, 52·55%. M.p. 115° F.
Sweating.
Nos. of fractions 1 2 3 4 5 6 1 2 3 4 5 6
Period sweating .. .. hours 6 6 6 6 6 — 6 6 6 6 6 —
Temperature of oven .. °F. 110 115 120 125 130 residue (yellow). 100 105 110 115 120 residue (white).
Melting-point of fractions °F. 95·5 111 115 120 125·5 139 95 100·5 106 110·5 115 124
Percentage of fractions .. .. 0·87 2·59 2·43 2·05 3·87 35·54 4·12 2·99 4·52 9·21 14·44 17·26
Yields in one sweating by m.p.—
135° F. m.p. .. .. .. 30·43%
125° F. .. .. 45·16%
Sweats and soft, 95–111° F. m.p. paraffin .. .. 24·41%
100·00%
Yields in one sweating by colour—
White wax .. .. .. 17·26%
Quantity to be re-sweated, fractions 3, 4, 5, 6 and 5A 58·33%
Wax at this stage must be pure white after decolorizing treatment.
9·67
7·59
49·08%. 135
18·65%. 135
30·43%. 135
45·16%. 125

Diagram B.

Total Scale = 100 %.—M.p. 120·5° F.



Yields in two sweatings by m.p.—

135° F. m.p.	43·89%
125° F. m.p.	29·88%
Sweats and soft paraffin, 89–113° F. m.p. ..	26·23 %
	100·00%

Yields in one sweating by colour—

White wax	41·80%
Quantity to be re-sweated, fractions 4–7	42·44%

Diagram C.

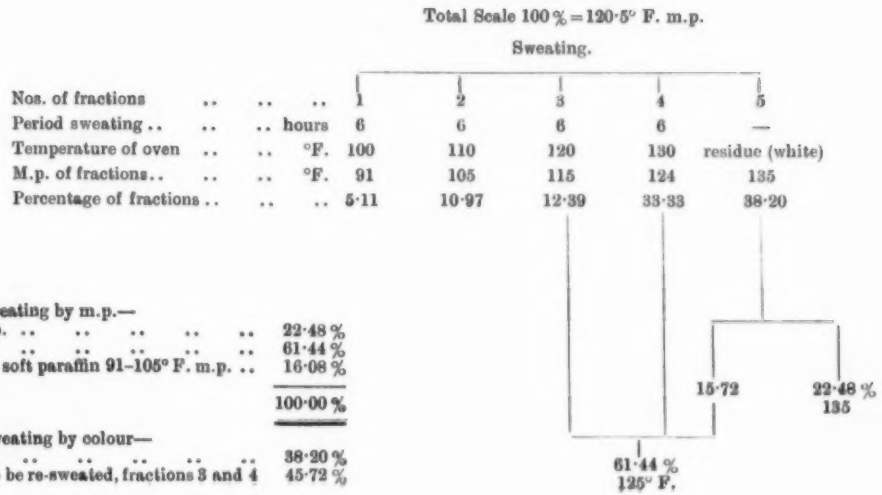
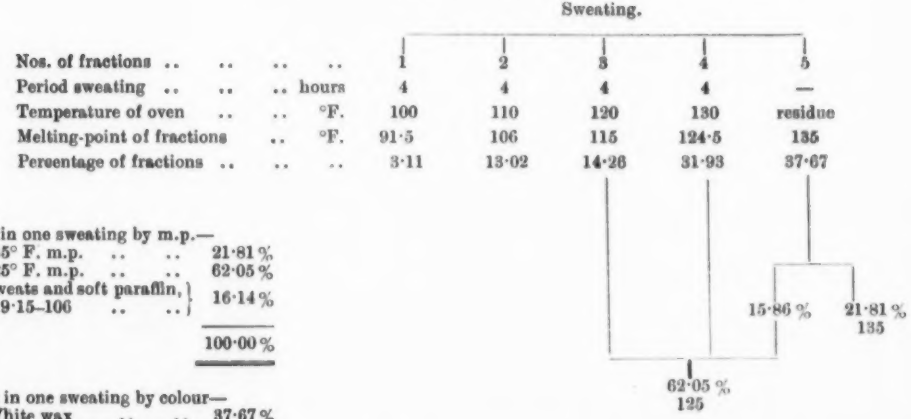

Total Scale 100 % = 120·5° F. m.p.
Sweating.
Nos. of fractions 1 2 3 4 5
Period sweating .. .. .. hours 6 6 6 6 —
Temperature of oven .. .. °F. 100 110 120 130 residue (white)
M.p. of fractions .. .. .. °F. 91 105 115 124 135
Percentage of fractions .. .. .. 5·11 10·97 12·39 33·33 38·20
Yields in one sweating by m.p.—
135° F. m.p. .. .. 22·48 %
125° F. ,, ... 61·44 %
Sweats and soft paraffin 91–105° F. m.p. .. 16·08 %
100·00 %
Yields in one sweating by colour—
White wax .. .. 38·20 %
Quantity to be re-sweated, fractions 3 and 4 45·72 %
15·72
22·48 %
135
61·44 %
125° F.

Diagram D.

Total Scale 100 % = 120·5° F. m.p.


Sweating.
Nos. of fractions 1 2 3 4 5
Period sweating .. .. .. hours 4 4 4 4 —
Temperature of oven .. .. °F. 100 110 120 130 residue
Melting-point of fractions .. °F. 91·5 106 115 124·5 135
Percentage of fractions .. .. .. 3·11 13·02 14·26 31·93 37·67
15·86 %
21·81 %
135
62·05 %
125

Yields in one sweating by m.p.—

135° F. m.p.	21·81 %
125° F. m.p.	62·05 %
Sweats and soft paraffin, 9·15–106	16·14 %
	100·00 %

Yields in one sweating by colour—

White wax	37·67 %
Quantity to be re-sweated, fractions 3–4	46·19 %

only 42·24 % required re-sweating. When this was done a further quantity, practically 32 %, reached the white stage.

As will be observed, these experiments were carried out in stages of 6 hours, sweating at each rise of 5° F. in the temperature of the oven. By comparison of the time occupied in each case, 30 hours sweating in A yielded only 41·2 % of the white product yielded in 42 hours in B.

That is to say, with an apparatus containing 100 % of scale divided in two parts as in A, 30 hours were taken to sweat the residues to 135·5° F. and 125° F. respectively—the latter only being white—while the same size of apparatus working the same quantity of scale as in B produced more than double the yield of white wax in 42 hours.

We then proceeded to ascertain if a similar result was obtainable in a shorter period. Diagrams C and D show that when the periods were reduced to 6 hours, and ultimately 4 hours' duration, with rises of 10° F., yields similar to those in B were obtained. We had therefore succeeded in reducing the total period for satisfactory sweating to 16 hours, as compared with the unsatisfactory treatment of the scales separately lasting 30 hours. Consequently, considering all these results conjointly, with the apparatus described could be obtained similar results to the lengthy sweating in B, by working the first stage as in D for 16 hours, and the second stage as in B for 18 hours.

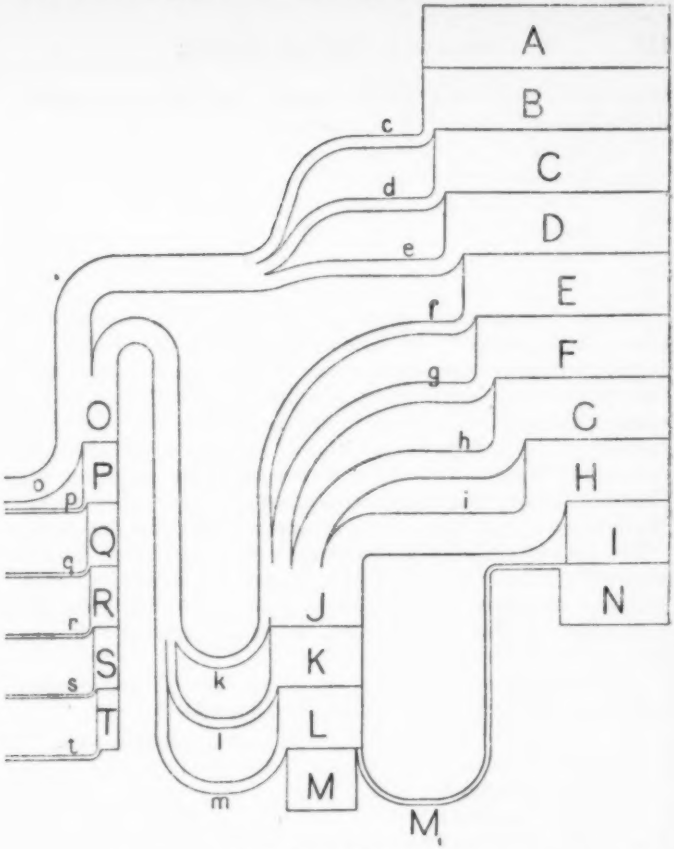
It will be seen from diagram B that when the sweatings from the first and second stages were mixed, crystallised, and again sweated, 6·5 % melting at 122·5° F. was recovered, although not white in colour. The fractions sweated out at this stage were more or less oily. The following are in our opinion the conclusions to be drawn from these experiments.

1. Sweating of the total scale produced a much greater yield of white paraffin in less time than the treatment of the hard and soft scales separately. While we recognise the necessity for special experiment with the scale produced from different crude oils, we are inclined to think that the same result will obtain generally, provided the distillates are of good quality, and the melting-point range of the scale similar to that used in our experiments.

2. The efficiency of the fractional sweating depends on the efficiency of the apparatus employed. One type of apparatus, worked by both the above methods, will bear the same relation to each other as our experiments, and give comparative results.

It has already been mentioned that the chief use to which paraffin wax is put is the manufacture of candles. Frankland [11] experi-

FIG. 1.—Sweating of Paraffin Scale: temperature increased 5° F. every 6 hrs.



FIRST STAGE SWEATING.

A, Total Scale 100%, M.P. 120·5° F.: B, Stove Temperature 100° F.: C, 105° F.: D, 110° F.: E, 115° F.: F, 120° F.: G, 125° F.: H, 130° F.: I. Residue White Wax from First Stage Sweating 41·80 %, M.P. 135·5° F.

c–i. Sweats from First Stage Sweating. c, 4·07 %, M.P. 89° F.: d, 5·55 %, M.P. 101·5° F.: e, 6·13 %, M.P. 104° F.: f, 5·46 %, M.P. 110·5° F.: g, 7·83 %, M.P. 116° F.: h, 12·37 %, M.P. 121° F.: i, 16·78 %, M.P. 124·5° F.

MIDDLE SWEATS (f, g, h, i = 42·44 %) CRYSTALLISED AND RESWEATED.

J, Stove Temperature 105° F.: K, 110° F.: L, 115° F.: M+M_1, Residue White Wax from Second Stage Sweating, 29·88 % = 2·09 % + 31·97 %, M.P. 125° F.

k–m. Sweats from Second Stage Sweating. k, 4·39 %, M.P. 100·5° F.: l, 2·80 %, M.P. 106° F.: m, 3·30 %, M.P. 113° F.

SOFT SWEATS (c, d, e, k, l, m = 26·24 %) CRYSTALLISED AND RESWEATED.

O, Stove Temperature 90° F.: P, 95° F.: Q, 100° F.: R, 105° F.: S, 110° F.: T, 115° F.

Residue Wax (coloured) from Third Stage Sweating 6·53 %, M.P. 122·5° F.

o–t. OILY SWEATS, 19·70 %, FROM THIRD STAGE SWEATING RETURNED TO PROCESS OR BURNED AS FUEL.

o, 11·10 %, M.P. 84° F.: p, 1·35 %, M.P. 92° F.: q, 1·13 %, M.P. 97° F.: r, 1·11 %, M.P. 101·5° F.: s, 1·52 %, M.P. 107·5° F.: t, 3·50 %, M.P. 112·5° F.

WHITE WAX. I + M_1 = 43·89 %, M.P. 135° F.

mented with illuminating value of various materials, and assessed their values as follows:—

Young's Paraffin Oil	1 gallon.
American Rock Oil No. 1	1·26 „
Paraffin Candles	18·6 lbs.
Sperm „	22·9 „
Stearine „	27·6 „
Tallow „	36·0 „

Another comparison[13] of the illuminating value of stearine, paraffin, and composite candles is shown in the table opposite.

Paraffin wax becomes plastic under the influence of gentle heat, that distilled from lignite being, according to Gräfe[14], superior for candle-making to that obtained from petroleum. This defect is partially neutralised by the addition of stearine, which, although reducing the luminosity to some extent, raises the bending-point. Montan and Carnauba waxes are also used for the same purpose. We have made some experiments in connection with bending-point.

The materials, paraffin and stearine, were moulded into candles measuring $7\frac{3}{4}$ in. long and $\frac{11}{16}$ in. diameter, and exposed to a gradually increasing temperature until the candle bent from the perpendicular. The period at each temperature-rise of 5° F. was 30 minutes.

Experimenting with paraffin wax melting at 133° F. and stearine melting at 130·5° F. the results were as follows:—

	Melting-point.	Bending-point.
100% Paraffin ...	... 133	... 93
80% Paraffin, 20% Stearine ...	... 129	... 108
70% Paraffin, 30% Stearine ...	... 127	... 109
60% Paraffin, 40% Stearine ...	... 124	... 115

Using 139° F. m.p. paraffin, and 130·5° F. m.p. stearine we obtained the following results:—

	Melting-point.	Bending-point.
100% Paraffin ...	... 139° F.	... 110
80% Paraffin, 20% Stearine ...	... 135·5	... 119
70% Paraffin, 30% Stearine ...	... 133·5	... 120
60% Paraffin, 40% Stearine ...	... 133·0	... 125

General Properties

Sort of Candle.	Size and Weight.			Wick.			Melting-point.	Solidifying-point.	Material burnt in 1 hour.	Height of Flame.				Light Intensity A.N.K.*			Material burnt per hour in A.N.K.	1,000 grms. material produce A.N.K. hours.
	Length.	Diameter.	Average weight.	No. of threads.	No. of fibres.	Weight of 1 m. of wick.				Largest.	Smallest.	Medium.	Most general.	Maximum.	Minimum.	Average.		
Stearine candles—	cm.	cm.				gr.	°C.	°C.		mm.	mm.	mm.	mm.					
1	28·6	2·0	79·1	5	25	0·908	54–55	56–55	8·5766	50	47	48·6	48	1·18	1·01	1·09	7·868	127·0
2	27·5	2·0	77·5	5	25	0·868	46–47	50	8·5510	48	45	46·9	47	1·16	1·04	1·11	7·704	129·8
3	24·9	1·7	51·1	5	25	0·875	46–48	50–46	8·7187	45	40	42·0	44	1·08	0·82	0·91	9·581	104·4
4	25·7	2·0	74·9	3	36	1·293	50–51	51·5–50·5	9·6368	51	48	49·6	50	1·28	1·20	1·23	7·835	127·6
5	26·4	2·0	75·9	3	36	1·362	47–49	48–47	9·4560	54	48	51·3	50	1·38	1·22	1·28	7·387	135·4
6	19·0	2·4	77·86	5	60	2·155	55–56	58–57	10·8989	73	57	66·8	67	2·10	1·60	1·79	6·088	164·2
7	22·6	2·2	79·2	5	55	2·063	56–57	58–57	10·5260	66	59	62·0	62	1·46	1·22	1·30	8·097	123·4
8	25·6	2·1	78·6	5	45	1·58	54–55	56–55	9·8436	60	53	56·3	57	1·20	1·08	1·13	8·711	114·8
9	28·0	2·0	78·1	3	34	1·24	50–51	53·5	9·5755	57	52	54·4	55	1·33	1·13	1·24	7·722	129·5
10	29·6	2·1	96·1	5	50	1·68	51·5–53	58–56	10·3594	63	57	59·3	60	1·40	1·13	1·32	7·848	127·4
Paraffin candles—																		
11	24·0	2·0	70·3	3	42	1·135	56	54	8·4684	63	55	60·1	60	1·50	1·23	1·35	6·273	159·4
Composite candles—																		
12	29·5	2·1	92·9	3	45	1·78	47–48	44	8·5356	58	53	55·8	56	1·32	1·11	1·23	6·939	144·1

* "A.N.K." = Amyl acetate normal candle.

When paraffins of differing melting-points are mixed, the melting-point of the mixture is the average of those mixed, but when mixed with stearine the m.p. is frequently below that of either constituent. The following further illustrates the effect of such mixtures:—

Parts Stearine, m.p. 130° F.		Parts Paraffin Wax.	Melting-point of mixtures with:— 120° F. m.p.	126° F. m.p.	127° F. m.p.
55	...	45	114	113·5	115·5
60	...	40	119	116	118
65	...	35	121	118	120
70	...	30	122·5	122·5	122
75	...	25	124·5	124	124
80	...	20	125·25	125·5	125·5
85	...	15	126	127	127
90	...	10	127·5	128	129·5
95	...	5	128·5	129	130

The tendency of paraffin wax to become discoloured on exposure to sunlight was commented on by Sommer[15], who expressed the opinion that this was due to the presence of unsaturated hydrocarbons. As these are present to a greater or less extent in all heavy oil and paraffin distillates, it is consequently important that, if wax is to be produced which will not discolour rapidly under these conditions, the sweating operation should be conducted thoroughly. The final treatment is also of great importance in this respect, for some decolorising mediums, bauxite for instance, possess the selective property for absorbing unsaturated bodies, and traces of the latter left in the sweated wax may thus be reduced, if not entirely removed.

The colouring of candle materials in various tints is mostly done with aniline dyes. These are in some cases insoluble in paraffin, but soluble in stearine; consequently the method of procedure is to dissolve the dyes in a portion of stearine, which is added to the mixture in sufficient quantity to give the desired tint.

The transparency or opacity of paraffin wax has received some attention, and different explanations of the cause of the variation in its appearance have been given. Neustadtl[16] states that transparency occurs only in wax composed of constituents not differing widely in melting-point. Mittler[17] contradicts this statement, asserting that opacity is due to the presence of oil to the extent of not less than 0·5 %. By mixing transparent paraffins of m.p. 108° and 135° F. respectively, he obtained a product which was also transparent.

Sommer [15] states that transparent paraffin has a formolite number not above 0·3, whereas the opaque variety will give a number as high as 1·6. As these figures are indicative of unsaturated hydrocarbons, which must be present as oil, these results are in agreement with those of Mittler. A contradictory statement is made by Singer[16], who found that the content of oil in samples of opaque and transparent paraffin may be approximately the same, and in one case a sample of transparent paraffin contained 3·6 % of oil. The higher melting waxes may be rendered transparent by subjection to gentle pressure and heat. Transparency is probably due to the state of crystallisation, and absence of air between the crystals.

Paraffin contracts to a great degree on solidification, according to Gräfe [17], 11–15 % and even more in the case of wax of very high melting-point. A paraffin melting at 111° F. sp. gr. in the solid state 0·867, and showing a contraction of 11 % on solidification, thus has a sp. gr. 0·771 at its melting-point, which, on the basis of a temperature-coefficient of 0·0004, represents a theoretical sp. gr. at 60° F., the liquid state being maintained, of 0·791. Similarly paraffin of melting-point 136° F. sp. gr. when solid 0·915, and contraction on cooling 15 %, has sp. gr. at its melting-point 0·7775, equivalent to 0·808 at 60° F. The sp. gr. 0·791 to 0·808, average 0·800, applies to ordinary paraffins at temperatures not much above their melting-points, and is also found to be applicable to paraffins dissolved in oil, the paraffin behaving as an oil of sp. gr. of 0·800.

In a paper on petroleum refining [20] read to you nearly two years ago, various apparatus for the manufacture of paraffin wax were described. Those for extraction were the inventions of Henderson, Bryson, and Thomson. The earliest method of separating hard scale was to allow the distillates to cool to atmospheric temperature in shallow pans. The solidified mass was then shovelled into canvas bags, which were tied up, and thrown into a perforated chamber to drain, after which the contents were pressed in hydraulics to remove as much oil as possible. The filtrate was then refrigerated to a lower temperature to recover the softer paraffin. One of the first devices for artificially cooling oil and paraffin was the drum cooler [21]. Cold brine was circulated through a horizontal drum revolved in a tank into which the oil to be cooled was pumped continuously. The oil solidified on the surface of the drum, and was removed by a scraper, falling into a trough from which it was pumped to the filter press. Cooling was very rapid, and the separation in the filter press less thorough than is the case when

cooling takes place slowly, as it does in the modern inventions. Another apparatus was a cylinder cooled externally by brine circulated from a refrigerator, and fitted with scrapers in which the contents were cooled to any given temperature [23]. This is the basis of all modern cooling apparatus; for instance, the Henderson cooler is a brine-cooled tank of such construction that a maximum cooling area is presented to the consecutive quantities of oil with which it is filled, while the various designs of continuous coolers obtain the same effect by passing the oil through in a constant supply.

The output of a cooler depends on the conditions under which it is worked. For example, a Henderson cooler puts through 2,400 gallons cooling from 86° F. to 35° F. in 24 hours with brine at 13° F. The time for pumping off through filter presses is included. In cooling average paraffin distillate from 120° F. to 40° F., 1,500 gallons can be cooled and put through in the same period, brine temperature 16° F. Other types of coolers show similar variation. The viscosity of the oil in which the paraffin is contained is, of course, an important factor to be reckoned with in connection with filtration.

An apparatus differing in principle from any of the aforementioned is the design of Beilby, which relates to apparatus for cooling and crystallising paraffin oils in a state of repose. The oil is filled into the cells, and allowed to remain quiescent until the desired temperature is reached. A rotating screw or helical cutter is provided at the bottom of each cell, and by operating this the frozen mass is forced into the filter pump. Sir Boverton Redwood [23] gives details of working this cooler. A charge of oil of temperature 100° F. was reduced to 15° F. in 72 hours by brine of temperature 10° F., and it is stated that "the crystallisation of the paraffin was so perfect that the separation of the oil was comparatively easy, and the filter presses were filled up every three hours as in the old drum-cooling process." For special purposes a cooler of this type might be very suitable, but for ordinary work its capacity is too limited.

The sweating process for refining crude scale was patented by John Hodges [24] in 1871, and the following abstract from the specification makes that gentleman's claims quite clear, also the general procedure in paraffin refining previously to that date.

"Heretofore crude paraffin has ordinarily been purified by allowing it to crystallize slowly after being melted and by subsequently pressing the crystallized cake so obtained to express out from amongst the crystals of paraffin the fluid and more easily melted

portions with which they are mixed. By this process, however, a quantity of the fluid and more easily melted portions still remain enclosed amongst the crystals of paraffin.

"To further purify the paraffin the operation is repeated, and in some cases heavy and light oils have been mixed with the paraffin when melted, and before allowing it to crystallize, in order to facilitate the subsequent expressing out of the fluid and more easily melted portions. In this case the paraffin after being pressed has required to be subjected to distillation to distil off the lighter oils remaining in it. This process is expensive and occupies much time.

"According to this invention the crude paraffin having, if desired, been first separated from rough impurities is cast into cakes, and allowed as heretofore to cool slowly so as to form into well-developed crystals. The cakes are then placed on a bed of absorbent or porous material (at present we find dry sand covered with some thin woven fabric or with filter paper to answer well) and are then, while thus placed, exposed to a warm temperature sufficient to render fluid the more easily melted portions with which the crystallized paraffin is mixed, but insufficient to melt the paraffin. The fluid and more easily melted portions then flow out from between the crystals of paraffin and into, or into, and through the absorbent, or absorbent and porous materials on which the cake rests. The cake thus freed from the fluid, or fluid and more easily melted portions, may then be treated in any usual method to render it white or as much lighter in colour as may be practicable, or the process may be repeated until the separation of the harder from the softer portions has been effected, as completely as is desired, and then any requisite process for whitening the product may be adopted if needed. The softer portions may be recovered by any suitable and usual process from the absorbent or porous bed."

In the actual operation of his process, as set forth in the patent specification of the above date, Hodges heated the ovens containing the sweating racks with air heated to the required temperature by blowing it over a steam coil. Subsequently sweating developed into laying the crude scale cakes, weighing about 10 lbs. each on corrugated iron shelves covered with coir matting arranged conveniently on iron racks inclined at an angle sufficiently great to admit of free drainage. The rooms containing these were heated with steam coils, which arrangement is still in use. Now, for quantities to be handled by large producers, such a process involves a vast amount of labour. Take for instance a refinery making 50 tons daily of total scale, 40 % to be re-sweated. The convenient

size of cake is, as said above, 10 lbs.; 50 tons of crude scale represent 11,200 cakes to be removed from the cooling shed to the sweating room, and 40 % re-sweatings add 4,480 cakes to this load, making 15,680 10-lb. cakes in all to be removed from the racks to the melting boiler.

Among several modifications of Hodges' process which were suggested, the following may be mentioned. In Tervet and Allison's [25] arrangement the melted crude paraffin was run into pans upon bands of cotton canvas. After solidification the bands with the hardened scale on them were drawn into a sweating stove. They also invented a system of trolley racks the trays of which were filled with the scale in a cooling room. After solidification these were moved on rails into the sweating room, the sweated product being removed after the rack was withdrawn. Tervet [26] patented an invention for sweating the scale in vertical cells. The scale was run into moulds, and when solidified lowered into a sweating cell constructed of gauze or perforated metal, and situated below. When sweating was completed, the product was dropped into the space underneath this cell. The patent of James and William Baxter [27] was a mechanical contrivance for sweating the cakes of scale placed on edge inside perforated cases, these being fixed to parallel endless chains mounted on suitable shafts. On completion of sweating, movement of the chains inverted the cases, emptying the sweated paraffin into a receptacle below.

None of these inventions effected labour saving as do Henderson's tray [28], and subsequently the cylinder form [29] of sweating stove. In these the scale is filled and removed in the liquid state, necessitating only a minimum of labour for attention, and obviating entirely the laborious transfer by hand of the crude scale cakes to the sweating stove, and the removal of the product to the melting boiler. It is curious to note that one of the most recent patents reverts to the trolley system, and heating with air blown over a steam coil [30].

In America, the greatest wax-producing country in the world, the Henderson system has been generally adopted, but in the Whiting plant of the Standard Oil Co., sweating is carried out in large cylindrical tanks containing as much as 40,000 gallons [22]. The tank is heated with hot-water pipes erected vertically, and the scale surrounding these sweats out the oil and low-melting-point scale as the temperature is increased. These collect on horizontal plates provided with run-off connections. A double sweating takes place, and the residue is finally melted in the usual way.

Following are examples of sweating in the tray form of stove.

A. Sweating a mixture of hard and soft scale :—

Melting-point of mixture	... ...	127° F.
Time of cooling	... ...	24 hours
„ „ sweating	... ...	21 „
Yellow wax as residue	... ...	47·2% m.p. 133° F.
Sweatings, yellow	... ...	43·8% „ 117
Soft sweatings and oil	... ...	9·0% „ 100

B. Similar mixture sweated until residue was white :—

Melting-point of mixture	... ...	126–127° F.
Time of cooling	... ...	24 hours
„ „ sweating	... ...	35 „
White wax as residue	... ...	31·5% m.p. 138° F.
Sweatings, yellow	... ...	59·5% „ 120° F.
Soft sweatings and oil	... ...	9·0% „ 100
		100·0

Generally speaking, the tray form of sweating stove requires about 58 hours for the complete cycle of operations, the length of time for cooling or sweating varying with the composition of the contents, thus :—

M.p. of mixture, ° F.	Time for filling.	Time for cooling.	Time for sweating.	Time for melting.
127 ...	½ hour	21 hours	17 hours	3½ hours.
117 ...	½ „	31 „	17 „	2½ „
108 ...	½ „	28 „	11½ „	2½ „

Following are examples of sweating in cylindrical stoves :—

A. Melting-point of scale ... ... 111° F.

Time of cooling	... ...	12 hours
„ „ sweating	... ...	48 „
White wax residue	... ...	33·3% m.p. 127° F.
Sweatings, yellow	... ...	31·4% „ 113° F.
Soft sweatings and oil	... ...	9·0% „ 100° F.

B. Melting-point of scale ... ... 107° F.

Time of cooling	... ...	28 hours
„ „ sweating	... ...	40 „
White wax residue	... ...	45·0% m.p. 124° F.
Sweatings, yellow	... ...	25·0% „ 113° F.
Soft sweatings and oil	... ...	30·0% „ 89° F.

It might be worthy of attention here to note that Hodges stated that better separation resulted in sweating when the scale was kept for some time after solidification. It has been suggested

that it is unnecessary for this period to elapse before heating is commenced, thereby shortening the time of operation and increasing the capacity of the plant; and this we submit is a point which might repay investigation. In other words, fractional crystallisation arrested at a certain point, allowing the unsolidified portions to drain off before fractional sweating is commenced, may be a means of economy in time, and consequently in plant. We may mention that experiments carried out by us lead us to believe that such procedure would be quite practicable and beneficial on the large-scale operation of cylindrical stoves.

The following somewhat novel suggestion was recently made for refining scale, and an Indian patent [2] was granted to R. S. Dickie for "improvements in and relating to a process of separating wax scale from petroleum and apparatus therefor."

"This invention relates to a process and apparatus for extracting and purifying wax scale in the petroleum refining industry, and is an improvement on what is known in the art as the sweating process. In that process, in order to remove the heavy oil from the scale, it is stacked in stoves on racks which are arranged at a suitable angle. The temperature of the stove is then slowly raised to that at which the heavy oil sweats out from the scale, the oil flowing away by gravitation owing to the angle in which the racks are placed. This sweating process is expensive in fuel and wasteful in time, the stacking and heating of the stove, and the sweating out of the oil, requiring as much as from 6 to 8 hours.

According to this invention the hard, middle and soft scale, or a mixture of all three grades, is treated and converted into yellow wax in a single operation in the following manner. The material to be treated is melted at as low a temperature as possible and is then pumped through a spraying nozzle, where it is acted upon by a blast of cold air which atomizes and chills the scale, the temperature of the material when issuing from the nozzle being from 5 to 10 degrees Fahrenheit below its melting point. Atomization of the scale at the nozzle takes place at a temperature low enough to solidify the wax, but not the oil and low-melting-point paraffin, which must remain liquid. The atomized mass passes from the nozzle into a rapidly revolving drum, the periphery of which is formed of a fine-mesh wire cloth, and the liquid portion is driven by centrifugal force through the interstices of the wire cloth, into an annular trough arranged outside the drum. From one to three minutes suffices for the separation of the heavy oil from the scale.

The residue of yellow wax at this stage, not being sufficiently liquid to be thrown off through the wire cloth, adheres to the interior of the drum. The drum is, therefore, then heated electrically to about 140° F., at which temperature the wax melts, and is driven off through the wire cloth into a second annular trough which takes the place of the first-mentioned or oil-recovering trough."

While it is apparent that this inventor is comparing his process with the sweating of small cakes, and not with the stoves of the Henderson type, it will be agreed that the application of centrifugal

motion and electrical heating to paraffin refining is a novel departure. Details of working results have not, as yet, been published.

Another departure from established methods for extracting and refining paraffin is that suggested by Edeleanu[38]. This is claimed to be "a method of separating the solid paraffin hydrocarbons from a mixture containing the solid paraffin hydrocarbons in association with liquid paraffin hydrocarbons, in which the mixture of hydrocarbons is treated with a sufficient quantity of the unsaturated hydrocarbons of the aromatic series, dissolved in sulphurous acid to dissolve the liquid paraffin hydrocarbons." It remains to be proved, however, that this process has practical application, and in the event of its success fractionation would still require to be carried out to produce grades of different melting-points.

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[A full Bibliography would require more space than can at present be accorded to it. That subjoined consists of additions to the entries in "Redwood," ed. iii.]

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DISCUSSION.

The President, in opening the discussion, said he was afraid the paper did not admit of very much controversy, since it dealt mainly with facts. The only point to which he wished to draw

attention in connection with the uses of paraffin wax was that the authors had omitted to refer to its use in the manufacture of chocolate cream, for which he believed it was largely employed. No doubt it was out of consideration either for the manufacturers of sweets, or for the ladies who eat them, that the authors had failed to make any reference to that point.

Sir Boverton Redwood, Bart., said the paper was unquestionably one of an exceptionally instructive character, its authors being accredited masters of the art with which it dealt. As far as he was aware a great part of the information given in the paper had never hitherto been accessible, and it could not fail to be of great value to those who were concerned in the technology of paraffin-refining. As an object lesson in respect of the use to which it could be put for propaganda purposes he was sure it would appeal to the Honorary Editor, Mr. W. H. Dalton, as illustrating at any rate one branch of the work of the Institution which he had consistently advocated, viz.: the diffusion of knowledge in relation to matters with which the members were intimately concerned, and which, but for the existence of the Institution, might never have been disclosed. He thought the authors would probably agree with him that, of the various types of cooler which had been described and illustrated, that which had been referred to as the Thomson cooler might be accepted as the latest development of the art of cooling an oil containing solid hydrocarbons with the object of effecting their separation. That type of cooler was somewhat of an innovation, because until recent years it had been considered that in order to obtain what were termed bold crystals in a crystallised mass from which the oil could subsequently be separated with facility, slow cooling of undisturbed bulk was essential to success. But apparently the Thomson cooler, which was now employed by the Oakbank Oil Co., appeared to be superior in regard to economy and efficiency to the older type of apparatus employed for that purpose. Quite recently his old friend Mr. John Wishart, the Managing Director of the Oakbank Oil Co., was good enough to give him a short description, in the following words, of the latest form of that particular type of cooler: "It has been designed for economy in the cooling tower, and to allow a larger extraction of paraffin at the first cooling of the heavy oil and paraffin from the second distillation, as well as from the final stage. The heavy oil and paraffin is first run through a tower, meeting a current of air from a small blower, which cools the heavy oil and paraffin to a temperature which shows crystals of paraffin in the liquid. Then it passes through an annular water cooler fitted with scrapers;

afterwards through a similar vessel cooled by the cold oil from the filter-presses, and finally it is pumped direct through the anhydrous-ammonia cooler to the filter-presses. The ammonia cooler is also subject to a gradually diminishing temperature as the heavy oil and paraffin passes from the top inlet to the bottom outlet."

An interesting use of paraffin wax to which he might be allowed to refer, was in the construction of models of ships to be tested by traction in such tanks as the Froude tank at the National Physical Laboratory, the existence of which they owed to the generosity of Sir Alfred Yarrow. It was found in effecting the necessary very slight alterations in the contour of the models during the process of testing them (in order to decide upon the best lines for the vessel ultimately to be constructed on the basis of the models) that paraffin was a very convenient material to employ, on account of the facility with which it could be moulded, and subsequently altered in contour by shaving.

He had been much impressed with the diagram that had been shown indicating the manner in which the process of sweating, or fractional fusion, progressed with the gradual improvement in the colour and melting-point of the material. He hoped it would be possible to reproduce that drawing in the journal of the Institution, as he felt sure it was well worthy of being rendered available for future reference. He thought the Institution was to be congratulated on having induced the authors to make such an exceedingly valuable contribution to the literature of the subject, and the least the members could do was to show their appreciation of it, in the usual manner, when an opportunity was afforded at a later stage.

Mr. Lawson Lomax was sure the members of the Institution felt very much indebted to the authors for the results of the excellent series of experiments on the sweating of paraffin scale for the production of white wax which had been placed before them. Those who had been engaged in that particular branch of the petroleum industry had felt the need of such information, and had looked in vain for publications dealing with the subject. He was particularly interested in the results given on the different methods of sweating, i.e. the merits of sweating the hard and soft scales separately or mixed in the proportions as pressed from the oil. The figures given showed the absolute superiority of the results obtained by sweating the mixed fractions as against the results obtained when sweating the fractions separately. In that connection he remembered some time ago, when having comparatively large stocks of yellow wax on hand, he was in urgent need of further supplies of white wax

The yellow wax was not amenable to decolorising earths, and recourse was had to re-sweating. The cakes were therefore re-sweated in the old-type cake-sweating room, and although the original colour of the wax was lemon yellow, yet it was necessary to sweat away between 60–70 % of that hard wax before the residue was sufficiently improved to give a white wax on treatment with char. It seemed therefore that for successful sweating a fairly wide range of fractions was necessary, together with slow raising of the temperature. Possibly in the case he referred to the addition of sweats to the hard wax before re-sweating would have resulted in a greater total yield of white wax.

Mr. H. V. Mitchell thought that one rather fundamental matter in connection with paraffin wax production was well worth additional mention, viz., the distillation of the astatki, or heavy oil fraction, in order to obtain the "heavy oil and paraffin." It was vital that the circumstances governing that part of the wax manufacture should be carefully studied, as endless trouble and delay in the subsequent filtering and sweating processes was incurred whenever tarry matter, in consequence of irregular distillation, found its way into the distillate. Moreover, it was very difficult, if not even quite impossible, to decide by the usual tests, or by appearance, whether the H. O. & P. distillate had suffered, and the trouble became apparent only when the sweating stage was reached. In vacuum distillations generally, the benefit of the vacuum was felt only when high vacua were available, in other words it was the last inch that counted. On the other hand, it was a difficult matter upon a large bench of stills to maintain a high and steady vacuum, whereas the tendency towards "bumping," which liquids boiling under low pressure always exhibited, was present in a fairly marked degree, even though the vacuum might be only partial. For that reason, on a bench where the vacuum pumps were incapable of maintaining a high vacuum, and where, moreover, the steam superheating arrangements were badly designed, he had favoured dispensing with the vacuum entirely. In that way the character of the distillate at the open worm ends could be seen at a glance, and at the first appearance of contamination the distillate could be diverted to a residue of fuel tank. Nevertheless, he felt that high vacuum conditions, together with a carefully regulated supply of perfectly dry steam, would give the best results. With a good vacuum more use could be made of dephlegmation without abnormal firing. As the success of the whole of the subsequent refining operations was dependent upon the quality of the H. O. & P., he felt sure that details as to size of

stills, dephlegmation, and state of vacuum, would be highly instructive and useful.

Dr. Dunstan said that although he would not presume to criticise in any shape or form the technology of the paper, there were a few minor accidental, rather than incidental, points of chemical interest to which he desired to be allowed to refer. He noticed that the author quite early in the paper made a reference to ceresin. In connection with this substance it should be noted that Marcusson had recently suggested that it differed from the ordinary wax of commerce, in being a mixture of isoparaffins rather than normal paraffins. Unfortunately the complexity and the extremely resistant nature of these substances rendered analysis and the application of chemical reactions very difficult.

One would rather imagine that the method of Dickie would be inoperative, seeing that the atomising of the melted wax would result in extremely rapid cooling, and therefore would produce crystals possibly much too small for any subsequent purification. He would like to know if the authors had had an opportunity of seeing that process at work, and could give any particulars as to how the scale compared with that produced under ordinary conditions.

Some very considerable doubt seemed to exist with regard to the physical constants of the normal paraffins. For instance, F. Kraft, who distilled paraffin from lignite, quoted the paraffin $C_{36}H_{74}$ as having a melting point of 76° C.; whilst Mabery gave the same melting point for $C_{35}H_{72}$. A series of such hydrocarbons should be produced synthetically, so that the physical constants could be known once and for all. Anyone who had ever carried out experiments on the combustion of such hydrocarbons knew the difficulty of deciding whether in fact a paraffin was being dealt with, or a substance with two atoms of hydrogen less.

The authors had also referred to the discovery of paraffin in Derbyshire, and in that connection he desired to refer to the paper of J. B. Cohen and C. P. Finn on "Paraffins in a Yorkshire Coal Seam" (1912). Those two investigators isolated paraffin which analysed to $C_{20}H_{42}$, $C_{22}H_{46}$, $C_{24}H_{50}$, $C_{25}H_{52}$, $C_{26}H_{54}$, and $C_{28}H_{58}$, together with substances of b.p. 160°–266°, which behaved as paraffin.

An interesting point raised by the authors was why paraffins crystallised better after being distilled. This might possibly be explained as being another illustration of the colloidal state of petroleum. It was a well-known fact that many colloids could be coagulated by the effect of heat, and this was possibly a question

of that nature. If heating was carried out to an excessive extent, paraffins cracked very easily indeed.

Another point of distinct interest was the coloration of paraffin wax when exposed to sunlight and atmospheric oxygen. A great amount of work had been done on that subject. For instance, in 1917 Schmidt heated paraffin wax to moderate temperatures, exposed it to air, and obtained a 70 % yield of acids. Again, Bergmann in 1918 heated Galician wax with a melting point of 52° C. to 130° C. for 15 days, and found that it gave a brown mass with an acid value of 132, and that it contained lignoceric acid and isopalmitic acid. These facts, the speaker thought, might explain the discolouring effect of light, i.e. that the light accelerated the ordinary effect of oxygen. He mentioned further that if vaseline was heated in the presence of alkali, the hydrocarbons could be turned into a soap. Paraffins were by no means so non-reactive as was generally supposed.

The final point he desired to refer to was the expulsion from scale of the least trace of oil, which the authors seemed rather inclined to regard as being unsaturated. The only criticism he could make in regard to that was that the oils that Mabery expressed and found associated with the definite paraffins he isolated were naphthenic in type, and saturated.

Mr. E. A. Evans said that it appeared to him that the unsaturated hydrocarbons which are found in the higher distillates of petroleum are not so readily oxidised when alone as is generally supposed. True, Mabery (*Journ. Amer. Chem. Soc.*, xxxix, 2028, 1917) found that the unsaturated hydrocarbons which he isolated from gilsonite by distillation, and subsequent extraction with sulphuric acid, freely absorbed oxygen from the atmosphere, producing a dark-coloured liquid, which finally became viscous. The resulting substances were found to contain oxygen. Mabery records the fact that unsaturated hydrocarbons do not exist in light-coloured varieties of petroleum, such as Berea Grit or West Virginia crudes. In this connection it is interesting to note that Mr. Evans has found that an oil, containing possibly 15 % of material abstracted with strong sulphuric acid, gave no solid oxidation-products when air was bubbled through the oil, even in the presence of metallic copper, for 45 hours at 150° C., whereas another oil, with a similar absorption with sulphuric acid, gave 0·7 % of solid matter under exactly similar conditions. If these oxidation-products are entirely produced from the unsaturated hydrocarbons, why the difference in the two results? It was thought that the sulphur-content might have had an influence, but in each case it was found to be about

0·03 %. Mabery's unsaturated hydrocarbons were free from sulphur. Evidently there is some unknown factor which governs the oxidation of petroleum hydrocarbons, and gives rise to discolorations and solid deposits.

Mr. A. Campbell, in reply, said the authors agreed that the Thomson cooler referred to by Sir Boverton Redwood was a very good apparatus, but the preliminary cooling arrangements that were referred to might also be applied to other forms of coolers. They were applied to work in connection with the Henderson cooler. That was a point that had been omitted from the paper.

The authors agreed with the remarks that had been made about the range of the melting point. It was necessary to get the range, otherwise the effect of sweating would not be obtained. That was one of the main points to bear in mind.

With reference to Mr. Mitchell's question in regard to the vacuum, good paraffin scale would not be obtained if good distillates and a good distillation were not employed. If what was known as a bad "back end" of the distillation was obtained, it was necessary to re-distil it, and bring it up to the point of the good portions before it was of any use to put it through the paraffin extraction plant. It was common practice at the present time to run heavy oil and paraffin with a good supply of dry steam, not particularly superheated, with 20–22 in. of vacuum, and obtain excellent results right up to the end of distillation.

The other points that had been raised were more or less academic, and required a considerable amount of research to clear them up. With regard to decoloration, very excellent results were obtained from paraffin which was properly treated. It was of no use to make bad distillates and bad paraffin scale, and then expect the decolorising treatment to atone for all the errors that had been made. The whole process must be watched from beginning to end, and if that was done, and the paraffin worked up to the point indicated in the diagram, most excellent results were obtained by filtration through bauxite.

On the motion of **The President,** a very hearty vote of thanks was accorded to the authors for their valuable contribution to the proceedings of the Institution, and the meeting terminated.